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OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

OFFICE OF PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: DDVP (dichlorvos) Registration Standard

FROM: Charles L. Trichilo, Chief
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

TO: Amy Rispin
Science Integration Staff
Hazard Evaluation Division (TS-769C)

and

George LaRocca
Product Manager (15)
Insecticide/Rodenticide Branch
Registration Division (TS-767C)

Attached are the Product Chemistry and Residue Chemistry Chapters for the DDVP Registration Standard prepared by Dynamac Corporation and secondarily reviewed by Debra F. Edwards. The due date for these chapters is August 29, 1986. This Standard includes data available and reviewed up to August 15, 1985.

As required by the Registration Standards Policy Group, the Product Chemistry Data for end-use products are not included in this Standard. This "generic" Standard is concerned with data which are relevant to technical formulations and manufacturing-use formulations containing DDVP. Also, as requested by Herb Harrison of the Registration Division, the Residue Chemistry Branch no longer addresses the physical/chemical properties of manufacturing-use producs. These will be considered later by the Registration Division as manufacturers respond to the "data call-in" program.

Product Chemistry data pertaining to end-use formulations are unique to each different formulation of DDVP and as outlined in 49 FR No. 207, p. 42856, October 24, 1984, must be submitted prior to reregistration and will be reviewed by the Registration Division as part of the reregistration process.

The Product Chemistry Chapter contains Appendix A and B in which is listed Confidential Business Information and is to be protected. Only those copies of the Standard in RCB and those sent to George LaRocca and the Toxicology Branch contain such information.

If you need additional input, please advise us.

cc: Anne Barton, HED (cover memo only)
Irving Mauer, TOX (with CBI Attachment)
Judy Heckman, HED (cover memo only)
John Jordan, EAB
T. Johnson, EEB
Kyle Barbehenn, SIS

bcc: Subject File
Reading File
Registration Standards File (with CBI)
Debra F. Edwards (with CBI)
PMSD (ISB)

TS-769:RCB:D.Edwards:2/21/86:R.Schmitt:2/26/86:Rm-810:CM#2:557-7484

DYNAMAC
CORPORATION

Final Report

DDVP
Task 2: Residue Chemistry Chapter

Contract No. 68-02-4266

January 28, 1986

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
Enviro Control Division
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DDVP

RESIDUE CHEMISTRY

Task 2

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DDVP

RESIDUE CHEMISTRY

TASK 2

INTRODUCTION

DDVP (2,2-dichlorovinyl dimethyl phosphate) is an insecticide federally registered for use on greenhouse-grown food crops including cucumbers, lettuce, mushrooms, radishes and tomatoes.

DDVP formulations are also registered for use on nonperishable bulk stored raw agricultural commodities (including animal feed, beans [dried typed], cocoa beans, coffee beans, grain crops such as corn, nut crops, peanuts, peas [field], soybeans and tobacco), and nonperishable packaged or bagged raw agricultural commodities (including beans [dried type], cocoa beans, coffee beans, grain crops such as corn, nut crops, peanuts, peas, and soybeans), and for direct use on dairy and beef cattle, poultry, goats, horses, sheep and swine. Additional use sites include ornamental plants and turf, domestic dwellings, noncrop aquatic areas, domestic pets, animal premises, eating establishments, and food processing and storage areas. DDVP formulations registered for use on greenhouse food crops, in mushroom houses, on raw agricultural commodities (nonperishable bulk stored, packaged or bagged), eating establishments (indoor edible), food markets, food processing, handling and storage plants/areas (indoor edible), and tobacco are: 0.0432% granular (G), 20% impregnated materials (Impr), 1-20% and 0.2-5.5 lb/gal emulsifiable concentrate (EC), 0.5-100% and 1.15-10 lb/gal soluble concentrate/liquid (SC/L), 0.5-20% and 0.74-5.8 lb/gal ready-to-use (RTU), and 0.25-40% pressurized liquid (PrL). DDVP formulations registered for use on tomatoes (postharvest application) are: 1% dust (D) and 4 lb/gal EC. DDVP formulations registered for use on animals and their premises are: 0.043-1% granular (G), 0.875 wettable powder

(WP), 0.5-4 lb/gal EC, 0.54-43.2% EC, 8-10 lb/gal SC/L, 0.25-50% SC/L, 0.194-1% RTU, 0.25% pelleted tablet (P/T), 20% Impr, and 0.5-1% PrL.

Numerous formulations contain DDVP as the sole active ingredient, or the formulations contain one or more active ingredients in addition to DDVP. For a complete listing of these formulations refer to the EPA Index to Pesticide Chemicals 2,2-dichlorovinyl dimethyl phosphate, dated 10/30/84.

Fumigation treatments are made to greenhouses using a thermal fog generator or other fogging devices while crops are growing. Mushroom houses are treated by fog application or by direct spray to surfaces. Nonperishable bulk stored packaged or bagged (raw agricultural commodities) can be treated by hanging insecticidal strips or by fogging application. According to the Preliminary Quantitative Usage Analysis of DDVP (J.E. Hogue, 1985, BUD, OPP, EPA), agricultural applications represent 60% of the total annual U.S. usage of DDVP. Of the total agricultural usage, less than one-half (25% of the U.S. total usage) is used for treatment of tobacco, greenhouses, and vegetables as well as sheep, poultry, and livestock other than dairy and beef cattle and swine. No details are available regarding use patterns involving specific crops. Tolerances for the food/feed items: cucumbers, lettuce, mushrooms, and tomatoes are currently expressed in terms of residues of DDVP (expressed as naled); tolerances for the remaining entries are currently expressed in terms of residues of DDVP [40 CFR 180.235 [a] and [b], 21 CFR 556.180, 21 CFR 558.205, 21 CFR 520.600, 21 CFR 193.140]. [NOTE TO P.M. - All tolerances for residues of DDVP should be expressed in terms of DDVP and not as naled.]

NATURE OF THE RESIDUE IN PLANTS

Conclusions:

The available data pertaining to metabolism of DDVP in plants are inadequate (refer to the Discussion of the Data). However, the available data pertaining to the metabolism of naled (1,2-dibromo-2,2-dichloroethyl dimethyl phosphate) in plants (Chevron Chemical Co. 1981.) are sufficient to describe the metabolism of DDVP (the initial, debrominated metabolite of naled).

Naled, applied directly to cotton and cucumber leaves, is metabolized primarily to DDVP which is quickly hydrolyzed (completely within 3 days) to dichloroacetaldehyde. Dichloroacetaldehyde is reduced to dichloroethanol which is then conjugated to form the 2,2-dichloroethanol glucoside and other carbohydrate conjugates (disaccarides and oligosaccharides). Refer to table 1 on p. 6 for a depiction of the molecular structure of DDVP and its metabolites. At the present time, the TOX Branch considers DDVP to be the sole residue of concern in plants (personal communication with J. Stewart, 2/24/86). However, it should be noted that additional data are being requested by the TOX Branch to support registration of products containing DDVP.

Tolerances for residues of DDVP in or on plant commodities are presently expressed in terms of naled (cucumbers, lettuce, mushrooms, and tomatoes) or DDVP (fresh and dried figs, and radishes). [NOTE TO P.M. - All tolerances for residues of DDVP should be expressed as DDVP, and not as naled. Thus, the tolerances for residues in or on cucumbers, lettuce, mushrooms, and tomatoes should be revised (expressed as DDVP).]

References (used):

00013545. Arthur, B.W.; Casida, J.E. 1957. Metabolism and selectivity of 0, 0-Dimethyl 2,2,2-trichloro-1-hydroxyethyl phosphonate and its acetyl and vinyl derivatives. Journal of Agricultural and Food Chemistry 5(3):186-191. (Also In unpublished submission received Apr. 16, 1965 under 5H1748; submitted by Shell Chemical Co.; CDL:22161-C.)

00074844. Casida, J.E.; McBride, L.; Niedermeier, R.P. 1961. Metabolism of 0,0-Dimethyl 2,2-Dichlorovinyl Phosphate (Vapona (R) or DDVP) in Relation to Residues in Milk and Mammalian Tissues. (Unpublished study received Aug. 20, 1962 under PP0330; prepared by Univ. of Wisconsin, Depts. of Entomology and Dairy Husbandry, submitted by California Chemical Co.; CDL; 090358-H.)

Chevron Chemical Co. 1981. [Ethyl-1-^{14}C]naled plant metabolism. Pages 6-30 In Metabolism chemistry data for Chevron naled technical (no MRID assigned).

References (not used):

[The following references duplicate information in previously cited MRIDs.]

00005296. Arthur, B.W.; Casida, J.E. 1957. Metabolism and Selectivity of 0,0-Dimethyl 2,2,2-trichloro-1-hydroxyethyl phosphonate and its acetyl and vinyl derivatives. Journal of Agricultural and Food Chemistry 5(3):186-192. (Report no. 1241; also In unpublished submission received Aug. 18, 1966 under 7F0612; submitted by Chemagro Corp.; CDL:090796-U.)

00059386. Casida, J.E.; McBride, L.; Niedermeier, R.P. 1961. Metabolism of 0,0-Dimethyl 2,2-dichlorovinyl phosphate (Vapona (R) or DDVP) in Relation to Residues in Milk and Mammalian Tissues. (Unpublished study received on unknown date under admin. no.; prepared by Univ. of Wisconsin, Depts. of Entomology and Dairy Husbandry, submitted by Shell Chemical Co.; CDL:120596-C.)

00117262. Casida, J.; McBride, L.; Niedermeier, R. 1961. Metabolism of ... Vapona or DDVP in Relation to Residues in Milk and Mammalian Tissues. (Unpublished study received June 28, 1961 under 201125; prepared by Univ. of Wisconsin, Depts. of Entomology and Dairy Husbandry, submitted by Shell Chemical Co., CDL:120058-A.)

Discussion of the data:

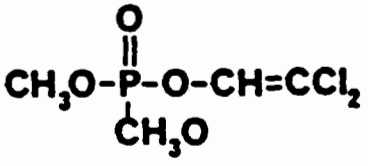
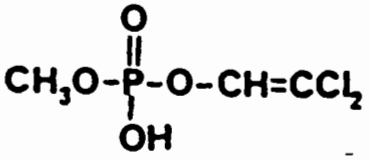
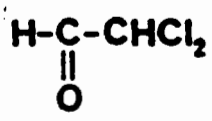
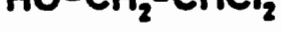
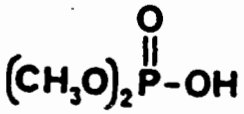
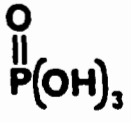
Arthur and Casida (MRID 00013545) examined the distribution of [^{32}P] DDVP in Perfection pea seedlings (of unreported age) incubated with their root systems in solutions of 35,000 cpm/ml for 24 hr. At the end of the exposure period, four plants were divided into root, stem, and leaf portions, and analyzed for total radioactivity by an undescribed method. Remaining plants were transplanted into silica sand and grown for 4 days at 25-30 C with no further pesticide treatment. At the end of the 24-hour exposure period, ^{32}P-residues were found in leaves and stems. No residue identification was attempted in this study.

Casida, et al. (MRID 00074844) administered [^{32}P]DDVP to 8-10 inch corn seedlings, 5 inch cotton seedlings, and 4-5 inch pea seedlings by uniformly applying 100 ul of an aqueous solution containing 0.1% [^{32}P]DDVP to the upper surfaces of the first three leaves (cotton only), and in other experiments by incubating seedlings for 8 hr with their roots in solutions containing 250 ppm [^{32}P]DDVP. Treated leaves from duplicate plants were collected at 0, 5, 10, 15, 20, 30, and 45 min., and 1, 2, 4, 8, 16, 24, 36, 48, and 72 hr. after treatment, washed two times with water to recover surface residues, and homogenized in water to recover absorbed residues. Plants receiving root treatments were washed with water, and incubated in distilled water for 40 hrs.; plants were collected for analysis at 0, 8, 16, 24, 32, and 40 hr. following termination of treatment, divided into leaves and stems/roots portions, and homogenized in water. For all samples, total ^{32}P-residues were determined by LSC and expressed as DDVP equivalents; organosoluble ^{32}P-residues were separated by chloroform/water partitioning prior to determination by LSC.

The authors report that approximately half of the foliar dose volatilized within 5 min of application to cotton leaves; an additional 45% was absorbed within 20 minutes. The surface residue was reported to be nonhydrolyzed DDVP (presumbly on the basis of ^{32}P-residues present in either the chloroform or the aqueous soluble fractions), while 70-80% of the absorbed material was organosoluble, thus likely hydrolyzed. Corn, cotton, and pea seedlings contained 27, 105, and 48 ppm ^{32}P-residues, expressed as DDVP equivalents after 8 hrs. of root exposure, respectively. Metabolites were not determined. [Naled hydrolyzes in aqueous solution (pH 7) with a $t_{1/2}$ of approximately 1.7 hours. Thus, the validity of these data is questionable since DDVP was applied and residues were extracted in water.]

The above studies do not adequately describe the nature of DDVP residues in treated plants. However, no additional data are required because the available data for naled (1,2-dibromo-2,2-dichloroethyl dimethyl phosphate), obained from the Naled Registration Standard (refer to Conclusions), provide an adequate understanding of the nature of DDVP residues in plants (DDVP = the initial, debrominated metabolite of naled).

Table 1. DDVP and its metabolites.

CODE	STRUCTURE	CHEMICAL NAME	ABBREVIATION; COMMON NAME
I		2,2-Dichlorovinyl dimethyl phosphate	DDVP; dichlorvos
II		2,2-Dichlorovinyl 0-methyl phosphate	DMD; desmethyl dichlorvos
III		2,2-Dichloroacetaldehyde	DCA
IV		2,2-Dichloro-1-ethanol	DCE
V		0,0-Dimethyl phosphate	DMP
VI		Phosphoric acid	inorganic phosphate

NATURE OF THE RESIDUE IN ANIMALS

Conclusions:

The available data pertaining to metabolism of DDVP in animals are inadequate (refer to the Discussion of the Data). However, the available data pertaining to the metabolism of naled (1,2-dibromo-2,2-dichloroethyl dimethyl phosphate) in animals (Chevron Chemical Co. 1982a and b; Chen. 1981) are sufficient to describe the metabolism of DDVP (the initial debrominated metabolite of naled). In poultry tissues and eggs, naled is debrominated to DDVP which is either hydrolyzed (major pathway) to dichloroacetaldehyde or O-demethylated (minor pathway) to desmethyl DDVP. Dichloroacetaldehyde is reduced to dichloroethanol or dechlorinated to glyoxylic acid. The dichloroethanol is conjugated with endogenous sulfate to form the sulfate ester conjugate of dichloroethanol. The glyoxylic acid is metabolized to glycine which is incorporated into proteins. Refer to Table 1 on p. 6 for a depiction of the molecular structure of DDVP and its metabolites.

The metabolism of naled in ruminants (goats) is similar to that in poultry. Naled is dehalogenated to DDVP which is hydrolyzed, resulting in residual fragments incorporated into amino acids, fatty acids, and sugars.

At the present time, the TOX Branch considers DDVP to be the sole residue of concern in animals and their products (personal communication with J. Stewart, 2/24/86). However, it should be noted that additional data are being requested by the TOX Branch to support registration of products containing DDVP.

References (used):

00013546. Casida, J.E.; McBride, L.; Niedermeier, R.P. 1962. Metabolism of 2,2-Dichlorovinyl dimethyl phosphate in relation to residues in milk and mammalian tissues. Journal of Agricultural and Food Chemistry 10(5):370-377. Also In unpublished submission received Apr. 16, 1965 under 5H1748; submitted by Shell Chemical Co.; CDL:221616-D.)

00066696. Potter, J.C.; Loeffler, J.E.; Collins, R.D.; et al. 1973. Carbon-14 balance and residues of Dichlorvos and its metabolies in pigs dosed with Dichlorvos-14C. Journal of Agricultural and Food Chemistry 21(2):163-166. (Also In unpublished submission received May 1, 1975 under 2724-269; submitted by Zoecon Industries, Inc.; CDL:220018-U.)

00117261. Block, A.; Panos, A.; Sheridan, J. 1961. Progress Report I on the Digestion of Dimethyl Dichloro Vinyl Phosphate in Cows: Project No. 9304-80-2. (Unpublished study received Sep. 6, 1961 under 5609-7; prepared by Evans Research and Development Corp., submitted by Norda Essential Oil & Chemical Co.; CDL:120055-A.)

00117262. Casida, J.; McBride, L.; Niedermeier, R. 1961. Metabolism of ... Vapona or DDVP in Relation to Residues in Milk and Mammalian Tissues. (Unpublished study received June 28, 1961 under 201-125; prepared by Univ. of Wisconsin, Depts. of Entomology and Dairy Husbandry, submitted by Shell Chemical Co.; CDL:120058-A.)

Chen, Y.S. 1981. Metabolism of [Ethyl-1-^{14}C]naled in a lactating goat. (Unpublished study submitted by Chevron Chemical Co; no MRID assigned.)

Chevron Chemical Co. 1982a. Metabolic fate of naled in chicken after a single oral dose of (Ethyl-1-^{14}C)naled. (Unpublished study; no MRID assigned.)

Chevron Chemical Co. 1982b. Characterization of ^{14}C in chicken tissues and eggs after dosing with (ethyl-1-^{14}C)naled for 10 consecutive days. (Unpublished study; no MRID assigned.)

References (not used):

[The following references were not used because they contain data/information which is irrelevant, illegible, or duplicates previously-cited references.]

00013545. Arthur, B.W.; Casida, J.E. 1957. Metabolism and selectivity of O, O-Dimethyl 2,2,2-trichloro-1-hydroxyethyl phosphonate and its acetyl and vinyl

derivatives. Journal of Agricultural and Food Chemistry 5(3):186-191. (Also In unpublished submission received Apr. 16, 1965 under 5H1748; submitted by Shell Chemical Co.; CDL:22161-C.)

00047474. Shell Chemical Company. 1970. Dichlorvos Metabolism. (Unpublished study received May 1, 1970 under 0H2477; CDL:221670-D.)

00066701. Loeffler, J.E.; DeVries, D.M.; Young, R.; et al. 1971. Metabolic Fate of Inhaled Dichlorvos in Pigs. Taken from: Toxicology and Applied Pharmacology 19:378 (Abstract no. 44). (Also In unpublished submission received May 1, 1975 under 2724-269; submitted by Zoecon Industries, Inc.; CDL:220018-AA.)

00074741. Casida, J.E.; McBride, L.; Neidermeier, R.P. 1961. Milk Residues following Oral Administration of Vapona or Dibrom to Cows. (Unpublished study received on unknown date under unknown admin. no.; prepared by Univ. of Wisconsin, Depts. of Entomology and Animal Husbandry, submitted by Shell Chemical Co.; CDL:120056-A.)

00074844. Casida, J.E.; McBride, L.; Niedermeier, R.P. 1961. Metabolism of O,O-Dimethyl 2,2-Dichlorovinyl Phosphate (Vapona (R) or DDVP) in Relation to Residues in Milk and Mammalian Tissues. (Unpublished study received Aug. 20, 1962 under PP0330; prepared by Univ. of Wisconsin, Depts. of Entomology and Dairy Husbandry, submitted by California Chemical Co.; CDL:090358-H.)

00116020. Shell Chemical Co. 1970. Residue Data Developed from the Use of Dairy Cattle: Rabon. (Compilation; unpublished study received Jan. 19, 1970 under 1F1090; CDL:090850-B.)

Discussion of the data:

Zoecon Industries, Inc. (MRID 00066696) submitted a study authored by Potter, et al (1973) in which nine Yorkshire cross male pigs received a single oral dose of [^{14}C]DDVP (labeled on the 1 position of the vinyl group) formulated as slow release polyvinyl chloride and administered at 40 mg/kg. Urine,

feces, and expired $^{14}CO_2$ were sampled at various intervals postdose and the animals (three per interval) were killed 2, 7, and 14 days posttreatment. Determination of total ^{14}C-activity was accomplished by liquid scintillation counting. A substantial portion of the administered ^{14}C-activity remained in the PVC pellets. Distribution of ^{14}C-activity is presented in Table 2. Of the administered dose, 91.2 to 98.2% was recovered from the group slaughtered 14 days postdose; material balance could not be calculated for the 2- and 7-day intervals because $^{14}CO_2$ from those groups was not monitored.

Table 2. Recovery of administered ^{14}C-activity (% of dose) at various intervals after treatment. Each range of recovery values represents three samples.

	2 days	7 days	14 days
Carcass	8.4-11.1	9.6-13.2	8.7-10.8
Feces	1.2- 3.4	3.9- 6.9	5.1- 6.3
Urine	2.4- 3.8	2.8- 3.3	2.4- 5.0
Expired $^{14}CO_2$	—	—	12.3-16.2
Voided pellets	31.8-38.0	45.6-46.6	62.7-64.9

^{14}C-Activity (expressed as DDVP equivalents) was detected in all tissues; the lowest levels (1.9-2.5 ppm) were found in the brain and the highest levels (9.7-32.9 ppm were found in the liver (Table 3).

Table 3. ^{14}C-residues, expressed as ppm DDVP equivalents, in tissues from pigs dosed with [^{14}C]DDVP at various intervals after administration.

	2 days	7 days	14 days
Gastrocnemius muscle	6.3	4.6	4.8
Quadriceps muscle	4.7	4.8	4.3
Mesenteric fat	2.6	4.2	2.5
Subcutaneous fat	1.6	4.0	2.2
Kidney	12.2	7.6	4.0
Liver	32.9	30.9	9.7
Other tissues/organs	2.5-12.3	1.9-8.0	1.9-5.6

Tissues were analyzed for residues of DDVP, desmethyl dichlorvos, dichloroacetaldehyde, and dichloroacetic acid using an undescribed GLC method; the authors report that no DDVP or any of its known metabolites were detected in any tissue. We find this study to be of little value because animals received only a single dose and were sacrificed in excess of 24 hours after treatment.

Norda Essential Oil and Chemical Co. (MRID 00117261) submitted a study prepared by the Evans Research and Development Corp. in which three cows received [^{3}H]DDVP as either an oral dose (90 mg in a gelatin capsule), intradermal injection (93 mg), or via a leg patch (66 mg). The label position was unspecified. Samples of milk, urine, and feces were collected at 12-hour intervals for approximately 3 days and total radioactivity in the samples was determined by liquid scintillation after wet digestion. Milk from the orally-dosed cow was separated into casein, fat, protein and whey fractions prior to ^{3}H-activity determination; 0.01 ppm, 0.01 ppm, and 0.22 ppm DDVP equivalents were detected in fat, protein and whey, respectively. No ^{3}H-activity was detected in casein fractions. Milk samples taken at 8-68 hours postdose contained total ^{3}H-residues (expressed as DDVP equivalents) at 0.25-0.37 ppm (oral dose), 0.14-0.18 ppm (intradermal dose), and 0.02-0.13 ppm (skin patch). We find this study to be of limited value because the [^{3}H]DDVP label position was not specified and residues in milk and tissues were not characterized.

Shell Chemical Co. (MRID 00117262) submitted a study conducted by Casida et al. which also appears in a published version (MRID 00013546) concerning the metabolism of [^{32}P]DDVP and/or [^{14}C]DDVP in cows, goats, and rats. Three Guernsey cows (one per dosing regime) received a single 1 mg/kg dose of [^{32}P]DDVP administered intravenously, subcutaneously or orally via capsule. A goat (breed unspecified) also received a single 1.52 mg/kg dose of [^{32}P]DDVP administered subcutaneously. Excretion routes of [^{32}P]DDVP in the test animals, expressed as a percentage of the administered dose were: subcutaneously dosed goat, 89% in urine, 11% in feces within 96 hours of treatment; intravenously dosed cow, 68% in urine, 13% in feces within 96 hours of treatment; subcutaneously dosed dow, 79% in urine, 15% in feces within 96 hours of treatment; orally dosed cow, 15% in urine, 53% in feces within 168 hours of treatment. Milk samples were taken at intervals from 0.25 hours to 144 hours postdose. Total ^{32}P-residues (expressed as DDVP)

peaked in 12-hour postdose milk samples from cows in all three administration routes. Total ^{32}P-residues in milk samples from the goat were 0.22 ppm at 0.25 hours, increased to a maximum of 1.82 ppm at 4 hours, and decreased to 0.20 ppm at 96 hours posttreatment. Milk samples were homogenized in chloroform, centrifuged, and and separated into chloroform-soluble, aqueous, and proteinaceous fractions. The protein fraction was further extracted with acetone and the resulting acetone fraction was combined with the chloroform fraction. Total ^{32}P-residues expressed as ppb DDVP present in the organosoluble fraction are summarized in Table 4. Further residue characterization in milk samples was not attempted.

Table 4. Organosoluble ^{32}P-residues (ppb) in milk from cows following administration of [^{32}P]DDVP at 1 mg/kg.

Hours Postdose	Intravenous	Subcutaneous	Oral
0.25	80	<15	1.5
0.5	38	<15	2.3
1	<10	<15	0.4
2	<10	<15	1.3
4	<10	<15	<1.0
8-96 (average)	<10	<15	<1.0

Female white rats received [^{14}C]DDVP labeled in the 1-carbon position at 4 mg/kg administered orally or intraperitoneally. Samples of urine and feces were taken at various intervals until sacrifice 7 days after dosing. Total radiolabeled desidues in urine, feces, and tissue samples were determined by liquid scintillation counting following homogenization of the samples in 0.015 N NaOH. Of the administered dose, 27-32% was eliminated via urine, and 3% via feces within 7 days after treatment; the authors also reported that considerable ^{14}C-activity remained in tissues, but percentages were not given. Urine samples were subjected to hydrolysis by refluxing in 8 N HCl for 6 hours prior to extraction. The presence of dichloroacetaldehyde (DCA) and a conjugate of dichloroethanol (DCE) (possibly dichloroethyl glucuronide) was reported, but no information regarding methodology was given. In another set of tests, male and female rats were orally dosed with [^{32}P]DDVP at 10-80 mg/kg. Dimethyl phosphate and inorganic phosphate were tentatively identified in rat urine samples based on cochromatography with known compounds on ion exchange columns.

RESIDUE ANALYTICAL METHODS

Conclusions:

Adequate GLC, spectrophotometric and Δ pH analytical methods are available for collection of data pertaining to residues to DDVP in plant commodities and animal tissues (refer to the Discussion of the data). For enforcement purposes, we recommend the use of GLC method I in PAM, volume II, reg. sec. 40 CFR 180.235. This method was originally published in JAOAC (1969. vol. 52, p. 1248), and subsequently submitted by Shell Chemical Co. (Method no. PMS-G-913-69; MRID 00049086). The method (slightly modified) underwent a successful method tryout on milk in 1971 (J. Mayes) and was validated to 0.01 ppm. Method modifications are included in the PAM. We recommend that GLC methods, preferably Method I in PAM vol. II, be used for data collection in the future. Although the colorimetric and Δ pH methods are acceptable for data collected in the past, these methods are nonspecific and, therefore, unsuitable for enforcement applications.

References (used):

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Discussion of the data:

A variety of methods was used for the generation of DDVP data. These include colorimetric procedures, GLC methods involving several different types of detectors capable of direct measurement of DDVP, a GLC method which measures DCA (a breakdown product of DDVP) and Δ pH methods. These methods involve various extraction and cleanup procedures before analysis can be accomplished. A GLC method submitted by Shell Chemical Co.(MRID 00049086) has been included in PAM, Volume II (Method I in Pesticide Reg. Sec. 180.235), with modifications, for enforcement purposes.

Shell Development Co. (MRID 00049086) submitted a GLC method, designated PMS-G-913-69, published by M.C. Ivey and H.V. Claborn (JAOAC 52:1248, 1969). Measurement of DDVP residues in or on crops using this method is accomplished by grinding or finely chopping a sample blended with CH_2Cl_2 and anhydrous Na_2SO_4. The extract is recovered by crop matrix filtration and concentrated to an appropriate volume by air blow-down. Milk samples are shaken with CH_2Cl_2 and Na_2SO_4, then filtered through fast-flow filter paper. Extracts are then evaporated by blow-down. Egg, animal tissues, and oily crop samples are

extracted with acetone instead of CH_2Cl_2, dried with anhydrous Na_2SO_4 and filtered through fast-flow filters. Aliquots must be cleaned-up by passage through minicolumns packed with polyethylene-coated alumina. The column eluate must be concentrated, extracted into CH_2Cl_2, concentrated again and exchanged with ethyl acetate. Modifications in extraction procedures for eggs, animal tissues and oily crop samples represent changes made to an earlier, nearly identical method submitted by the petitioner (PMS-G-913/68, MRID 00118113). Residues are detected by a GLC equipped with a thermionic detector (2% Reoplex 400 coated on 80/100 mesh GCQ). Recoveries of DDVP from two mushrooms samples fortified at levels of 0.1-0.2 ppm were 90-100% (Table 5). The reported detection limit is 0.002 ppm. This method is acceptable for both data collection and enforcement purposes. PMS-G-913-69 underwent a successful method try-out on milk (EPA memorandum dated 11/2/71 by J.E. Mayes; in correspondence file of PP#9F0788); the slightly modified method was found satisfactory for determining residues of DDVP in milk at a level of 0.01 ppm (modifications in PAM, vol. II, Method I). Method PMS-G-913/67 for determination of DDVP in sheep tissues (MRID 00115939) is a GLC method similar to those discussed above.

The U.S. Public Health Service, Food and Drug Administration submitted an unpublished modification of Method I in PAM, Vol. II (the "Dallas Method") developed by J.L. Power (MRID 00051556). In this method, unspecified meal samples are blended with water in a commercial blender or food processor, acetonitrile (ACN) is added to extract DDVP, and the mixture filtered through glass wool. A 10 g aliquot of the extract is transferred to a separatory funnel containing Na_2SO_4 and a mixture of petroleum ether and ethyl ether. The mixture is shaken and allowed to partition, and the ether layer removed. The combined ether extracts are then dried over Na_2SO_4 and concentrated to approximately 5 ml with a Kuderna Danish evaporator. The partitioned extract is then transferred to a 10 g florisil column, washed with 100 ml ethyl ether (discarded) and eluted with 2% MeOH in petroleum ether. The eluate is then evaporated to approximately 5 ml. Aliquots of the cleaned-up extract are then analyzed in a GLC equipped with a KCl_4 thermionic detector. This method, while providing satisfactory cleanup, resulted in significant sample losses. Most of the losses occurred during cleanup procedures, and

therefore, a published cleanup method (JAOAC 48:748), Boone's silicic acid column cleanup procedure, was tried and produced satisfactory recovery of DDVP. The petitioner slightly modified Boone's method for use with their meal samples. In this modification, DDVP is extracted from 100 g of the composite meal-H_2O mixture by adding Na_2SO_4, phosphotungstic acid and benzene to a centrifuge bottle. The bottle was thoroughly shaken and centrifuged at 1500 rpm for five min. The benzene extract was blown off, collected, and dried with Na_2SO_4. A second benzene extraction was similarly performed, extracts combined, and evaporated to approximately five ml. The concentrated extract was then passed through a silicic acid column, eluted with an unspecified solvent (see original "Dallas method"), and evaporated to five ml for GLC determination. The petitioner states that this Boone's modification is laborious and time-consuming. A third procedure, the Dallas Revised Method, was used successfully. In this procedure, sample preparation, ACN extraction, and the aliquot taken for partitioning are identical to those in the original method. The 10 ml aliquot of sample extract is transferred to a separatory funnel containing 2% Na_2SO_4 and petroleum ether, shaken vigorously for one min and allowed to separate. The water layer is transferred to a separatory funnel containing Na_2SO_4. The petroleum ether fraction is back-washed with ACN, the water layer added and the petroleum ether discarded. The aqueous fraction is shaken with two separate washes containing petroleum and ethyl ethers. The ether layer is then washed with Na_2SO_4 and concentrated to five ml. Aliquots of the concentrate are analyzed using a GLC equipped with a KCl thermionic detector. Recovery values and fortification levels are presented in Table 5 for all three procedures.

Shell Development Co. (MRID 00049087) submitted a GLC method, designated MMS-R-201, for determination of DDVP residues in animal feeds, blood, and urine; a subsequent submission by Shell Chemical Co. (MRID 00117257), designated MMS-R-201-1, outlined methods for determination of residues in turkey skin and lean tissue. Animal feeds are chopped or ground, an acetone-CH_2Cl_2 solution is added to the feed, and the mixture refluxed for 2-3 hr. CH_2Cl_2 is added in an amount equal to that lost during refluxing.

An aliquot of the extract is then centrifuged and a portion of the supernatant is diluted with CH_2Cl_2 to "obtain a chemical concentration of about 0.5 to 1.5 micrograms per milliliter". [How one would arrive at this concentration without knowing the DDVP concentration beforehand is unclear.] A portion of this extract is analyzed by GLC. Blood and urine are analyzed by adding a 1:1 solution of H_2O-absolute EtOH to the samples in a 2:1 ratio (solvent:sample). The solvent layer is then dried with anhydrous Na_2SO_4 and analyzed immediately or refrigerated until analyzed. Turkey skin samples are macerated and then extracted with ACN. The ACN extract is dried with Na_2SO_4, washed twice with hexane, and the pooled extracts concentrated. The hexane concentrate is then extracted with CH_2Cl_2 and the CH_2Cl_2 extract concentrated. Additional CH_2Cl_2 is added to tare and H_2O removed using Na_2SO_4. Calcium stearate and Columbia Carbon are added and the entire mixture is filtered. Aliquots of the filtrate are analyzed by GLC without further cleanup. Lean turkey tissue is chopped, CH_2Cl_2 added, and the mixture shaken to extract DDVP. The extract is then filtered over Na_2SO_4. Calcium stearate and Columbia Carbon are added, the mixture filtered and the filtrate concentrated by air blow-down. Aliquots of all final extracts are analyzed using a GLC equipped with a phosphorous detector. Recovery values were not reported for either method. The reported detection limit for MMS-R-201 is 0.25-5 ppm depending on sample type; that for MMS-R-201-1 is 0.02 ppm.

Shell Development Co. (MRID 00139845) submitted a GLC method, designated MMS-R-221-1, for detection of residues of DDVP in or on air, food and beverages. This method supercedes method MMS-106 (October 1968). Food samples are ground to a puree with H_2O. Food purees or beverages are mixed with Na_2SO_4, phosphotungstic acid and CH_2Cl_2. The mixture is centrifuged, if necessary, then dried with Na_2SO_4. Aliquots of the supernatant extract are analyzed by a GLC equipped with a phosphorus detector. Shell Chemical Co. (MRID 00118639) submitted a modification of this method, also designated MMS-R-201, for determination of DDVP residues in or on chicken tissues and eggs. In this method, 25-50 g of representative sample are extracted with ACN. ACN extracts (number not specified) are dried and an aliquot washed twice with

hexane to remove fats and other hexane-soluble coextractives. The washed ACN is concentrated, passed through a Snyder column and exchanged with hexane. An aliquot of the hexane extract is injected into a GLC equipped with an AFID phosphorus sensitive detector. The reported detection limit is 0.01 ppm. Reported recoveries were between 70-130% for 19 samples of egg, muscle and organ tissue samples fortified with 0.025-0.10 ppm DDVP (Table 5).

The U.S. Dept. of Agriculture (MRID 00060469) submitted a GLC method (method not given a numerical designation) for determination of DDVP residues in milk and body tissues of dairy cows treated for fly control. Milk samples are passed through a silicic acid column for cleanup. The solvent is a 3:2 mixture of CH_2Cl_2 and hexane. The eluate is condensed by passage through a Snyder column, hexane blown off and an appropriate aliquot injected into a GLC equipped with a flame photometric detector. Fat samples are extracted with hexane, partitioned into ACN and passed through a Snyder column. Repeated hexane washes are used to elute the samples. Muscle samples are extracted with a mixture of ACN and hexane. Extracts are equilibrated and the hexane phase is back-extracted with two portions of ACN. The ACN extracts are combined and run through a Snyder column which is eluted with hexane. Blood samples are blended with ACN, the extract solvent condensed, and ACN removed as described above for muscle and fat. Extracts of fat, muscle, and blood are cleaned-up by passage through a column containing Na_2SO_4 and silicic acid. The columns are eluted with CH_2Cl_2 and hexane. The solvents are removed from the column and the volume appropriately reduced. Extracts are analyzed using a GLC equipped with a flame photometric detector. Recoveries at a 0.01 ppm fortification level were 93-97% for milk, 80-85% for fat and muscle and 86% for blood (Table 5). The reported detection limits are 0.003 ppm in milk and 0.002 ppm in body tissues.

The U.S. Agricultural Research Service (MRID 00107572) submitted a GLC method designated PCB-69-8, developed by W. Jones, E. Miles and K. Hill (1968) for analysis of DDVP residues in or on tomatoes, radishes and land (water)

cress. This method is essentially identical to PCB-68-8 also presented in MRID 00107572 except that radishes, fruits and watercress are stored in Mason jars containing CH_2Cl_2 prior to analysis. Tomatoes are homogenized and blended with Na_2SO_4 and CH_2Cl_2, the homogenate filtered, the filtrate washed twice with CH_2Cl_2 and the CH_2Cl_2 filtrates combined. Other vegetables such as radishes, watercress and cucumbers are surface-stripped of residues with CH_2Cl_2. Samples are then homogenized and handled similarly to those from tomatoes. Extracts are cleaned up by adding Norite SG-extra and filtered through a column containing Celite and Na_2SO_4. Samples are eluted from the column with CH_2Cl_2, the filtrates combined and evaporated to 5 ml. Aliquots were analyzed with a GLC equipped with a flame photometric detector. Recoveries for samples of tomato fruits and foliage samples fortified at 0.04 ppm were 97 and 98%, respectively (Table 5).

Chemagro Corp. (MRID 00118115) submitted a GLC method, designated No. 27478, for determination of DDVP residues in or on bananas. Samples are extracted with a 2:1 H_2O-CH_3OH mixture. The extracts are centrifuged and partitioned with a 2:1 mixture of ether and petroleum ether. The combined solvent phases are dried and concentrated to 10 ml. Aliquots are chromatographed using a thermionic detector. The reported sensitivity of the method was 0.01-0.02 ppm. Recoveries from seven samples fortified with 0.05-0.1 ppm were 73-104% (Table 5).

A GLC method submitted by Oregon State Univ., Dept. of Agricultural Chemistry (MRID 00042706) and developed by U. Kiigemagi and L.C. Terriere is used for detection of residues in or on such crops as beans, broccoli, potatoes and cauliflower. This method is designated RM-3-C. To each sample, redistilled $CHCl_3$, anhydrous Na_2SO_4 and 4:1 H_2SO_4 are added. Samples are macerated in a blender for 5 min and the $CHCl_3$ fraction decanted and filtered through anhydrous Na_2SO_4. The $CHCl_3$ extract is placed in an Erlenmeyer flask along with 2 ml xylene and the mixture evaporated to approximately 5 ml under an air jet at about 50 C. The sample is then passed through an absorption column containing Na_2SO_4, silicic acid and Celite (Darco G60-Celite). The column is eluted

with 100 ml of equal parts diethyl- and petroleum ethers. One ml xylene and two ml glycerin in CH_3OH are added and the mixture is evaporated to ten ml in a rotary flash evaporator and concentrated to approximately one ml using an air jet. Aliquots are analyzed using a microcoulometric gas chromatograph. Reported sensitivities were 0.05-0.1 ppm. At unspecified fortification levels, recoveries averaged 79 and 84% from plant slurries and extracts, respectively (Table 5). In all cases, sample sizes were not reported.

Shell Chemical Co. (MRID 00049975) and Shell Development Co. (MRID 00042704 and 00060472) submitted similar GLC methods for analysis of crops and animal tissues, applicable for both dried and wet (>10% H_2O content) samples. These methods are designated PMS-G-900/66, MMS-50/64, and MMS-50A/64, respectively. In all procedures, samples are ground, chopped or macerated with H_2SO_4 and Na_2SO_4 is added to remove water. Samples are extracted repeatedly with ethyl ether. Combined extracts are filtered to remove particulates (wet samples are filtered over Na_2SO_4). Volumes are adjusted by evaporation or addition of ether to produce known sample concentrations. Detection is by GLC equipped with a phosphorous-sensitive electron capture detector. These methods measure dichloroacetaldehyde (DCA) instead of DDVP (the compound for which tolerances have been established). The sensitivity of all three methods is 0.01 ppm. No recovery values were given for any of the methods.

Shell Development Company (MRIDs 00042702, 00060470, 00074706, and 00140392) submitted four basic spectrophotometric methods for analysis of residues in or on crops and animal tissues. In method, MMS-58/64 (MRID 00140392), DDVP-treated samples are shaken with four volumes of H_2O using a mechanical shaker. An equal volume of CH_3COCH_3 is added, the mixture shaken for two hours and a portion of the CH_3COCH_3 centrifuged and filtered over Na_2SO_4. The filtered CH_3COCH_3 is placed in a flask, and an ethylene glycol:CH_3OH solution is added. The solvent is blown off and H_2O added. Five ml of resorcinol solution are added and the contents thoroughly mixed. Five ml of Na_2CO_3 solution are added and the solution completely mixed. The extract is then incubated at 55 C for 30 min, cooled and three ml of $NaHSO_3$ added. The volume is adjusted

to 25 ml and the absorbance vs. H_2O at 492 nm recorded. Cloudy samples require the addition of CCl_4. The DDVP concentration of unknowns is calculated from a standard curve. In enzyme inhibition method MMS-30/60 (MRID 00074706), butter, feeds, or animal tissues are chopped or ground with Skellysolve B and ACN. After vigorous shaking to thoroughly extract DDVP, the mixture is filtered, H_2O is added, and the mixture is extracted 4 times with petroleum ether. The combined extracts are then concentrated using a Snyder column. The extract is then diluted with CH_2Cl_2 and concentrated with dry air. The extract is cleaned up using the procedure described below. Crop samples are ground in a Waring Blender with CH_2Cl_2 and filtered and the filtrate is prepared as described below. Grains are extracted twice with CH_2Cl_2 and thereafter extracted in the same manner as for crops. Extracts of all types of samples are prepared by adding a 5% EtOH solution, and blowing off the CH_2Cl_2 by passing dry air through a capillary tube. Extracts are pipetted into cuvettes and turbid samples clarified by the addition of NH_4OH. Prior to incubation of unknowns, acetylcholine substrate and human blood serum reagents must be standardized and a standard curve constructed using analytical reagents. To each sample, HCl and $FeCl_3$ reagents are added. Analytical grade Celite is added to each sample and each is capped, vigorously shaken and filtered. The absorbance of the filtrate is measured at 540 nm. Method Pr 5e-62 (MRID 00049971) is essentially identical to MMS-30/60 except that flour, raisins and non-fat dry milk are placed initially in an extraction tumbler with CH_2Cl_2 for three hrs. Method DS.16.23.32 (MRID 00118592) duplicates MMS-30/60. Methods MMS-30/63 and MMS-30/65 are enzyme inhibition colorimetric methods which appear quite similar to MMS-30/60, but details were not available. Method MMS-30/64 (MRID 00042702) involves identical procedures for analysis of grains and crops as MMS-30/60. However, butter, feeds and animal tissues are extracted with a mixture of Skellysolve B and ACN. The extracts are filtered and the ACN boiled with hexane. The mixture is concentrated over a steam bath, additional hexane added and the eluate again concentrated to a small volume. By repeating this solvent exchange procedure an unspecified number of times, a complete exchange is effected. Five ml of the hexane extract is evaporated to 1-2 ml under a stream of dry air. Extracts are then prepared for analysis as described above. Method PMS-G-902/66 (MRID 00115993) is identical to MMS-30/64

except that EtOH is not used to solubilize lipids. Method MMS-30A/64 (MRID 00060470) outlines a different method of sample preparation for samples of butter, feeds and animal tissue. Samples are chopped and thoroughly blended with CH_2Cl_2 and Na_2SO_4. The mixture is centrifuged and the supernatant extract used in further cleanup and extraction steps. The final cleanup appears to be very similar to that described in method MMS-30/60. Method PMS-G-906/66 duplicates MMS-30A/64.

Chevron Chemical Co. (MRID 00074777) submitted an acetylcholinesterase inhibition-spectrophotometric method, designated RM-3a, capable of quantifying naled and DDVP (a breakdown product of naled), in samples containing both compounds. However, this method is not capable of distinguishing the two compounds. Details of the method for analysis of DDVP were not specifically stated but both plasma and bovine acetylcholinesterase enzymes are used. Four possible outcomes and their interpretations are:

1. No residues - no inhibition of either enzyme;
2. DDVP only - inhibition of plasma enzyme >> inhibition of bovine enzyme;
3. Naled only - inhibition of bovine enzyme >> inhibition of plasma enzyme;
4. Both residues - inhibition of both enzymes; in this case the amount of each depends on the relative concentration of naled and DDVP.

The petitioner states that this procedure is time-consuming and better suited for research applications than routine residue analysis.

Shell Chemical Co. (MRIDs 00117747) submitted a method for determination of DDVP residues (WAMS 30-1) and a modification of the method (also designated WAMS 30-1, MRID 00047472) based on accurate measurement of small changes in pH of solutions containing DDVP. In the modified method, samples of cheese and milk are macerated for two minutes with CH_2Cl_2 and Na_2SO_4, filtered and cleaned up by steam distillation. CH_2Cl_2 is removed by passing a stream of clean air through the aqueous solution. Both methods follow the sequence of steps outlined below. One ml of each solution to be assayed is placed into a reaction tube along with a CH_3OH solution containing glycerol. Any organic solvent present is evaporated using a stream of clean, dry air. Three ml of a plasma-saline-buffer solution are added to each reaction tube. Samples are incubated at 34 C. Blanks containing solvent instead of the pesticide inhibitor must be prepared identically to unknown samples. Thirty min after addition of enzyme, the pH of the contents of the tube is measured and one ml of acetylcholine chloride substrate is added. The pH of each solution is measured 60 min after the addition of the substrate. The percent residual activity is calculated using the expression:

$$R = 100x \frac{\Delta pH_i}{\Delta pH_b}$$

where ΔpH_i is the change in pH in the presence of inhibitor and ΔpH_b is the change in pH in the absence of inhibitor. The percent residual activity (R) is related to the percent inhibition (I) by the expression:

$$R = 100 - I.$$

Ruzicka et al. (MRID 05002074, 1968) submitted a published gel chromatography method for separation of solutions containing a variety of organophosphate pesticides. Sephadex LH-20 is allowed to absorb CH_3COCH_3, tetrahydrofuran and EtOH. Slurries are packed into glass columns and columns loaded

with one ml of the pesticide solution. Columns are eluted with the appropriate solvent at one ml/min. Fractions (size not defined) are analyzed using an unspecified GLC procedure. Method sensitivities and recoveries were not reported.

Keith et al. (MRID 05004395, 1968) submitted an NMR method for determination of organophosphorus pesticides including DDVP. However, details of the method are illegible and, therefore, cannot be summarized.

In summary, all methods submitted are adequate for data collection although we recommend use of GLC methods for future data collection. For tolerance enforcement, we recommend Method I in PAM, Volume II, Pesticide Reg. Sec. 180.215. Alternate GC columns, gel chromatography and mass spectrometry methods are also available for confirmatory procedures.

Table 5. Recoveries of DDVP from samples of fortified commodities.

Commodity	Method No.	Fortification (ppm)	Percent Recovery (Range)	MRID No.
Banana peel	27478	0.05-0.1	87-104(3)[a]	00118115
Banana pulp	27478	0.05-0.1	73-84(4)	00118115
Banana peel	MMS-30/60	0.05-0.2	80-85(3)	00118149
Banana pulp	MMS-30/60	0.05-0.2	79-84(3)	00118149
Banana peel	MMS-30/60	0.1	72(1)	00117689
Banana pulp	MMS-30/60	0.05-0.1	63-68(2)	00117689
Banana peel	MMS-30/60	0.1	85-90(2)	00058538
Banana pulp	MMS-30/60	0.1	76-80(2)	00058538
Beans	Pr 5e-62	0.116-2.32	78-98(6)	00056595
Bisquits	MMS-30/60	0.1	80(1)	00042707
Blood (cow)	Unspecified	0.01	86(N.R.)[c]	00060469
Cocoa beans	MMS-30/64	0.1-0.2	70-90(3)	00117747
Cocoa beans	MMS-30/64	0.1	75(2)	00117747
Cocoa beans	WAMS 30-1	0.04-0.2	95-100(3)	00117747
Cocoa beans	WAMS 30-1	0.05-0.1	76-81(2)	00117747
Corn (shelled)	Pr 5e-62	0.1-1.0	100-104(2)	00117747
Corn (shelled)	Pr 5e-62	0.1-1.0	100-104(2)	00117747
Chicken (breast)	MMS-30/64	0.1	100(1)	00116870
Chicken (breast)	MMS-30/60	0.05	77-102(3)	00116870
Chicken (drumstick)	MMS-30/60	0.05	50-102(2)	00116870
Chicken. (anus)	MMS-30/60	0.05	58(1)	00116870
Chicken (neck)	MMS-30/60	0.05	95(1)	00116870
Chicken (lean meat)	MMS-30/60	0.05	91(1)	00116870
Chicken (wings)	MMS-30/60	0.05	96(1)	00116870
Cucumbers	MMS-30/60	0.1	100(1)	00117686
Eggs (chicken - composite of one dozen)	MMS-30/64	0.1	95(N.R.)[c]	00116870
Eggs (chicken)	MMS-30/60	0.1	75-103(4)	00116870
Eggs (chicken)	MMS-30/60	0.05	82(1)	00116870
Eggs (chicken)	PMS-G-913/69	0.05	50-100(9)	00139841
Eggs	MMS-R-221-1 (modified)	0.025-0.1	80-120(7)	00118639
Egg yolk (chicken)	MMS-30/64	0.1	95(1)	00116870
Fat (chicken)	MMS-30/60	0.1	90(1)	00116870
Fat (chicken)	MMS-30/64	0.1	100(1)	00116870
Fat (chicken)	MMS-30/60	0.1	75-103(4)	00116870
Fat (chicken)	MMS-30/60	0.05	93(1)	00116870
Fat (chicken)	PMS-G-913/69	0.05	60-120(8)	00049085
Fat (chicken)	MMS-R-221-1 (modified)	0.05-0.1	70-130(2)	00118639
Fat and muscle (cow)	Unspecified GLC	0.01	80-85(N.R.)[c]	00060469

Table 5 continued.

Table 5. Recoveries of DDVP from samples of fortified commodities (continued).

Commodity	Method No.	Fortification (ppm)	Percent Recovery (Range)	MRID No.
Fat (subcutaneous, sheep)	PMS-G-913/67	0.2	75(1)	00115939
Figs	RM-3-G-3	0.005-0.25	80-88(3)	00076809
Flour	MMS-30/60	0.1	80(1)	00042707
Flour	Pr 5e-62	0.116-5.8	60-113(9)	00056595
Flour	WAMS 30-1	0.1	110-120(2)	00117747
Gizzard (chicken)	MMS-30/60	0.05	74(1)	00116870
Gizzard (turkey)	MMS-R-221-1	0.1	72(2)[d]	00139844
Gravy (unspecified)	MMS-30/60	0.1	80(1)	00042707
Heart (chicken)	MMS-R-222-1 (modified)	0.05	70-90(2)	00118639
Heart (pig)	MMS-30A/64	0.1	100-105(2)	00140392
Kidney (pig)	MMS-30A/64	0.1	100(2)	00140392
Kidney (sheep)	PMS-G-913/67	0.2	90(1)	00115939
Large intestine (pig)	MMS-30A/64	0.1	100(1)	00140329
Lean meat (chicken)	MMS-30/60	0.1	66-73(3)	00116870
Lettuce	MMS-30/60	0.1	102(1)	00033139
Lettuce	MMS-30/60	0.05	92(1)	00117683
Liver (chicken)	MMS-30/60	0.1	76(1)	00116870
Liver (chicken)	MMS-30/60	0.1	91-94(3)	00116870
Liver (chicken)	PMS-G-913/69	0.1-0.2	50-90(7)	00049085
Liver (chicken) (modified)	MMS-R-222-1	0.05	100-120(2)	00118639
Liver (pig)	MMS-30A/64	0.1	100(1)	00140392
Liver (sheep)	MMS-R-222-1	0.1	80(2)	00115939
Liver (turkey)	MMS-R-222-1	0.1	80(2)[d]	00139844
Lung (pig)	MMS-30A/64	0.1	90(1)	00140392
Meat (chicken - composite breast & leg)	MMS-30/64	0.1	100(N.R.)[c]	00116870
Meat (chicken)	PMS-G-913/69	0.05	70-100(2)	00049085
Milk (cow)	Unspecified GLC	0.01	93-97(N.R.)[c]	00060469
Milk (cow)	MMS-30/65	0.1	80-105(4)	00140392
Milk (cow)	MMS-30/63	0.1	70-80(3)	00140392
Milk (cow)	MMS-30/64	0.1	95-100(3)	00140392
Muscle (light, chicken)	MMS-R-222-1 (modified)	0.05	100-120(2)	00118639
Muscle (dark, chicken)	MMS-R-222-1 (modified)	0.05-0.2	80-110(3)	00118639

Table 5 continued.

Table 5. Recoveries of DDVP from samples of fortified commodities (continued).

Commodity	Method No.	Fortification (ppm)	Percent Recovery (Range)	MRID No.
Muscle (pig)	MMS-30A/64	0.1	105(1)	00140392
Muscle (sheep)	PMS-G-913/67	0.1	90(1)	00115939
Muscle (turkey)	MMS-R-222-1	0.1	98(2)[d]	00139844
Mushrooms	MMS-30/60	0.05	72-84(3)	00033142
Mushrooms	PMS-G-913-69	0.1-0.2	90-100(2)	00074658
Mushrooms	MMS-30/60	0.1	79(1)	00074658
Mushrooms	MMS-30/60	0.05	78-84(2)	00074658
Mushrooms	MMS-30/60	0.05	70-72(2)	00074658
Mushrooms	MMS-30/60	0.1	95(1)	00117686
Noodles	Pr 5e-62	0.1-5.0	80-115(8)	00056595
Omental fat (sheep)	PMS-G-913/67	0.05-0.1	96-100(2)	00115939
Ova (chicken)	MMS-R-222-1	0.05	110(1)	00118639
Peanuts	Pr 5e-62	0.3-5.0	67-92(7)	00056595
Plant slurries (unspecified)	RM-3-C	N.R.	84(N.R.)[c]	00042706
Plant extracts (unspecified)	RM-3-C	N.R.	79(N.R.)[c]	00042706
Potatoes	MMS-30/60	5.05-0.1	77-81(2)	00117687
Raisins	Pr 5e-62	0.116-0.58	69-81(4)	00056595
Renal fat (sheep)	PMS-G-913/67	0.05	90(1)	00115939
Rice	MMS-30/60	0.1	90-100(2)	00042707
Rice	Pr 5e-62	0.116-1.16	69 -101(5)	00056595
Skin (turkey)	MMS-R-221-1	0.1	71(2)[d]	00139844
Small intestine (pig)	MMS-30A/64	0.1	100(1)	00140392
Sorghum (grain)	Pr 5e-62	0.1-1.0	100(2)	00117747
Soybeans	Pr 5e-62	0.1-1.0	94-100(2)	00117747
Soybeans	PMS-G-913/69	0.1-0.2	60-120(18)	00117747
Spleen (pig)	MMS-30A/64	0.1	95(1)	00140392
Sugar	Pr 5e-62	0.116-2.32	69-94(6)	
Tomatoes (processed)	MMS-30/60	1.0	0.44	00141129
Tomatoes (processed)	MMS-30/60	10.0	35.8	00141129
Tomatoes (processed)	MMS-30/60	100.0	38.5	00141129
Tomato fruit	MMS-30/60	0.05	83(1)	00117686
Tomato fruit	MMS-30/60	0.1	95(1)	00117686
Tomato fruit	MMS-30/60	0.1	70-110(23)	00115993
Tomato fruit	MMS-30/64	0.04	97[b]	00107572

Table 5 (continued).

Table 5. Recoveries of DDVP from samples of fortified commodities (continued).

Commodity	Method No.	Fortification (ppm)	Percent Recovery (Range)	MRID No.
Tomato foliage	PCB-68-8	0.04	98[b]	00107572
Unspecified	MMS-30/60	0.01	100(1)	00118084
Unspecifed "meal" (solids)	Dallas Method	0.118	66-73(3)	00051556
Unspecified "meal" (liquids)	Dallas Method	0.059	80-103(3)	00051556
Unspecified "meal" (solids)	Boone's silicic acid cleanup of Dallas Method	0.059-0.118	52-111(9)	00051556
Unspecified "meal" (liquids)	Boone's silicic acid cleanup of Dallas Method	0.029-0.118	70-105(10)	00051556
Unspecified "meal" (solids)	Modified Dallas Method	0.059-0.118	75-95(9)	00051556
Unspecified "meal" (liquids)	Modified Dallas Method	0.059-0.118	83-108(4)	00051556

[a]Number of samples reported in parenthesis.
[b]Mean of an unreported number of samples.
[c]Number not reported.
[d]Mean of 2 samples.

STORAGE STABILITY DATA

Conclusions:

The available data are sufficient to ascertain that residues of DDVP in or on frozen plant samples are stable up to 90 days after application. Residues in or on frozen animal tissues are stable up to 8 weeks after application. The following additional data are required:

- o The storage intervals and conditions of storage of samples used to support all established tolerances for residues must be submitted. These data must be accompanied by data depicting the percent decline in residues at the times and under the conditions specified. (No additional stability studies are required for plant or animal commodities stored frozen for up to 3 months or 8 weeks, respectively). On receipt of these data, the adequacy of the aforementioned tolerances will be reevaluated.

- o All residue data requested in this standard must be accompanied by data regarding storage length and conditions of storage of samples analyzed. These data must be accompanied by data depicting the stability of residues under the conditions and for the time intervals specified, with the exception of plant commodities stored frozen for 3 months or animal commodities stored frozen for 8 weeks.

References (used):

00074776. Kohn, G.K. 1960. Letter sent to G.S. Hensill dated May 9, 1960: Storage Stability of Dibrom and DDVP in Fortified Extracts of Sorghum--Seeds and Heads. (Unpublished study received May 16, 1960 under 239-1281; submitted by Chevron Chemical Co; CDL:026977-A.)

00076809. Shell Chemical Company. 1980. Summary of Residue Data for Dichlorvos in or on Figs. (Compilation; unpublished study, including published data, received May 8, 1981 under 201-125; CDL:070073-A.)

00140392. Shell Chemical Co. 1971. The Results of Tests to Determine Whether Vapona Insecticide Residues Are Incurred in Food Products Derived from Livestock Treated with Vapona, Including a Description of the Analytical Methods Used. (Compilation; unpublished study received Sep. 8, 1969 under 9F0788; CDL:091359-A.)

Shell Chemical Co. 1967. Determination of Dichlorvos and Dichloroacetaldehyde Residues in Flour and Pinto Beans After Fortification. (Unpublished study submitted Apr. 6, 1967 under PP#7H2166; no MRID assigned).

Discussion of the data:

Chevron Chemical Co. (MRID 00074776) submitted a study conducted by G.K. Kohn of the California Spray Chemical Corporation concerning the stability of DDVP in or on sorghum seeds and heads. Extracts of both seeds and heads (solvent not specified) were fortified such that they bore an initial concentration of 0.05 gamma per ml. The recovery of DDVP from seeds at 0, 45 and 90 days after fortification was 93, 100 and 102%, respectively; recoveries of DDVP from seed heads at the identical intervals were 98, 102 and 102%. The conditions of storage were identified only as "stored frozen". The methods used to analyze the samples were not identified. No recovery or control values were included in the report, nor was it stated whether the submitted recoveries had been corrected by such values.

Shell Chemical Co. (MRID 00140392) submitted a study concerning the stability of DDVP residues in pig tissues. Frozen macerates of pig tissue including kidney, liver and muscle were fortified at 5 ppm of DDVP and stored up to 8 weeks at -25 C. Values extrapolated by eye from a graph of declines in

DDVP concentration over time were 5, 5.3, 4.7 and 4.2 ppm at 1, 2, 4 and 8 weeks, respectively (100, 106, 94 and 84% of initial values). The petitioner states that, based on these data, the half-life of the compound approximates 40 weeks. Such an extrapolation based on samples taken up to only 8 weeks must be considered tenuous. The method used to analyze the samples was not identified. No recovery or control values were included in the report nor was it stated whether the submitted recoveries had been corrected by such values.

Shell Chemical Co. (MRID 00076809) submitted data reflecting the stability of DDVP residues on figs. Samples were fortified with 0.005 to 0.05 ppm (theoretical), washed and dried at 48.9° C for 48 hours and stored frozen (exact temperature not specified) six weeks until analyzed. Uncorrected recoveries from two individual samples fortified at 0.025 and 0.05 ppm were both 100% and that from a single sample fortified at 0.005 ppm was 80%. Chevron method RM-3G-3 was used for analysis.

Shell Chemical Co. (no MRID assigned) presented data depicting the decline of DDVP residues in flour and pinto beans fortified with either 6.25 or 2.5 ppm of DDVP. Flour stored at ambient temperature (exact temperature not specified) and fortified with 6.25 ppm, bore residues of 6.25, 5.75, 4.6, 3.5, 1.4, 1.2 and 0.04 ppm at 0, 1, 2, 3, 6, 10, and 28 days, respectively, after fortification. Samples fortified at 2.5 ppm bore residues of 2.4, 1.9, 1.2, 1.15, 0.25 and 0.06 ppm at 0, 1, 2, 3, 7 and 10 days postfortification, respectively. The half-life of DDVP in flour fortified at 6.25 ppm was 4 days; that in flour fortified at 2.5 ppm was 2 days. Pinto beans fortified at 6.5 ppm and stored at ambient temperatures bore residues of 5.0, 3.5, 3.1, 2.3, 1.1, 0.8, 0.01 ppm at 0, 1, 2, 3, 7, 10, and 27 days, respectively. Samples fortified at 2.5 ppm bore residues of 2.1, 1.4, 0.72, 0.23, and 0.026 ppm at 0, 1, 3, 7, and 10 days, respectively. The estimated half-lives of DDVP in samples treated at 6.25 and 2.5 ppm are 3.5 and 1.5 days, respectively. The analytical methods used, recovery efficiencies and control values were not specified. These data suggest

that residues in or on plant commodities stored at ambient temperatures decline much more rapidly than those stored frozen.

MAGNITUDE OF THE RESIDUE IN PLANTS

It should be noted that the conclusions stated below are subject to change on receipt of the data requested in the section entitled "Storage Stability Data".

NOTE: Tolerances for residues of DDVP per se (40 CFR 180.235), and naled and DDVP (40 CFR 180.215) in or on mushrooms, cucumbers, tomatoes and lettuce are equivalent in magnitude. Thus, the registrant(s) must submit label amendments prohibiting the use of naled on crops which have been treated with DDVP.

Root and Tuber Vegetables Group

Conclusions for the Root and Tuber Vegetables Group:

A crop group tolerance is not appropriate at the present time because DDVP is registered for use only on radishes.

Radish

Tolerance:

A tolerance of 0.5 ppm has been established for residues of DDVP in or on radishes [40 CFR 180.235(a)].

Use directions and limitations:

The 10% RTU formulation is registered for fumigation (fogging applications) use on greenhouse-grown radishes at 16.7 fl oz of product per 50,000 ft^3. The 10% PrL is registered for fumigation use on greenhouse-grown radishes at 1 lb of product (0.1 lb ai) per 50,000 ft^3. Four applications may be made at three-day intervals, and the entire schedule may be repeated monthly. A 24-hour PHI is in effect.

Conclusions:

The available data are insufficient to assess the established tolerance for residues of DDVP in or on radishes because the maximum permissible number of applications was not reflected in the data. The following data are required:

- Residues of concern in or on radishes harvested 24 hours after the final application in a treatment regimen consisting of four fogging treatments at 3-day intervals repeated monthly and using either 16.7 fl oz of product of the 10% RTU formulation (applied with a thermal fog generator) or 1 lb product (0.1 lb ai) of the 10% PrL per 50,000 ft^3.

A Codex MRL of 0.5 ppm has been established for residues of DDVP in or on vegetables (except lettuce). No Canadian or Mexican tolerance has been established.

References (used):

00119536. Summit Chemical Co. 1969. Study: DDVP Residue in Tomatoes and Other Selected Crops. (Compilation; unpublished study received Sep. 25, 1970 under 6218-13; CDL:007833-A.)

00118572. Jones, W.; Miles, E.; Hill, K. 1970. Report of Residue Analysis: Dichlorvos. (Unpublished study received June 18, 1974 under 1327-36; prepared by U.S. Agricultural Research Service, Entomology Research Div., Pesticide Chemicals Research Branch, Analytical Investigations, submitted by Fuller System, Inc., CDL:024527-A.)

Discussion of the data:

Summit Chemical Co. (MRID 00119536) submitted data from a single MD test concerning residues of DDVP in or on greenhouse-grown radishes harvested

16-160 hours after application of a 10% aerosol (presumably a PrL formulation) released at 1 g ai/1,000 ft^3 (1.1x). Residues in or on seven radish samples, and in one untreated control were nondetectable ("0.00", limit of detection not specified). No recovery values were provided. Analyses were performed using GLC equipped with a flame photometric detector. Samples were stored for an unspecified period in methylene chloride, until analyzed. No data were submitted concerning residues in or on radishes following the maximum permissible number of applications.

Fuller System Inc. (MRID 00118572) submitted data from a single MD test concerning residues of DDVP in or on radishes harvested 16 to 350 hours after application (as a fog ignited from a fuel canister) of an unspecified 11% formulation released at 1.691 g/1,000 ft^3 (0.18 lb ai/50,000 ft^3; 1.8x). Residues of DDVP in or on radishes harvested 16, 40, 64, and 88 hrs posttreatment were 0.125, 0.079, 0.021, and 0.027 ppm, respectively. Residues of DDVP in or on six samples taken 112 to 350 hours after application were nondetectable ("0.00", limit of detection not quantified). Analyses were performed using a GLC equipped with a flame photometric detector. No residue was detected on one untreated plant control sample. Recovery efficiency of samples fortified at 0.02-0.5 ppm DDVP was 96% from pulp of "fruit and vegetables" (radish, however, was not specifically mentioned). Residue values were not corrected for recovery efficiency and control values.

The available data are insufficient to assess the established tolerance for residues of DDVP in or on radishes, since only one of four permissible applications was performed.

Leafy Vegetables Group

Conclusions for the Leafy Vegetables Group:

A crop group tolerance is not appropriate at the present time because DDVP is registered for use only on lettuce.

Lettuce

Tolerance:

A tolerance of 1 ppm has been established for residues of DDVP (expressed as naled equivalents) in or on lettuce [40 CFR 180.235(a)]. [Note: Tolerance should not be expressed as naled equivalents, but as DDVP per se.]

Use directions and limitations:

The 10% RTU formulation is registered for fumigation (fogging applications) use on greenhouse-grown lettuce at 16.7 fl. oz. of product per 50,000 cu. ft. using a thermal fog generator. The 10% PrL is registered for fumigation use on greenhouse-grown lettuce at 1 lb of product (0.1 lb ai) per 50,000 cu. ft. Four applications may be made at 3-day intervals and the entire schedule may be repeated monthly. A 24-hour PHI is in effect.

Conclusions:

The available data are insufficient to assess the established tolerance for residues of DDVP in or on lettuce because the maximum permissible number of applications was not reflected in the data and it was unspecified whether or not lettuce samples were analyzed with or without wrapper leaves. The following data are required:

- Residues of concern in or on lettuce, harvested 24 hr after the final application in a treatment series consisting of four fogging treatments at 3-day intervals repeated monthly during the growing season with either the 10% RTU formulation at 16.7 fl. oz. product per 50,000 cu. ft., or 1 lb product (0.1 lb ai) of the 10% PrL per 50,000 cu. ft. Application of the RTU should be made using a thermal fog generator. Residue data must be provided for lettuce with and without wrapper leaves.

A Codex MRL of 1 ppm has been established for residues of DDVP in or on lettuce. No Mexican or Canadian tolerance has been established.

References (used):

00033139. Shell Chemical Company. 1961. Residue Data for Vapona on Lettuce: RES-60-6. (Unpublished study received May 26, 1964 under 201-125; CDL:000814-A.)

00082271. Interregional Research Project Number 4. 1968. Residue Study of Dichlorvos on Tomatoes, Lettuce and Cucumbers. (Compilation; unpublished study received on unknown date under 0E0875; CDL:097531-B.)

00118572. Jones, W.; Miles, E.; Hill, K. 1970. Report of Residue Analysis: Dichlorvos. (Unpublished study received June 18, 1974 under 1327-36; prepared by U.S. Agricultural Research Service, Entomology Research Div., Pesticide Chemicals Research Branch, Analytical Investigations, submitted by Fuller System, Inc.; CDL:024527-A.)

00119536. Summit Chemical Co. 1969. Study: DDVP Residue in Tomatoes and Other Selected Crops. (Compilation; unpublished study received Sep. 25, 1970 under 6218-13; CDL:007833-A.)

References (not used):

[The following MRIDs were not used because they contain information which is irrevelant, illegible, or duplicates previously-cited information.]

00033144. Shell Chemical Company. 1963. Determination of Vapona Insecticide & Dichloroacetaldehyde Residues in Cucumbers, Lettuce, Mushrooms, Spinach & Tomatoes following Application of Vapona Insecticide: RES-63-116. (Unpublished study received May 26, 1964 under 201-125; CDL:000814-F.)

00107572. Jones, W.; Miles, E.; Hill, K. 1969. Report of Residue Analysis: PCB-69-8. (U.S. Agricultural Research Service, Entomology Research Div., Pesticide Chemicals Research Branch, Analytical Investigations; unpublished study; CDL:022406-A.)

00117683. Shell Chemical Co. 1961. Determination of Vapona Insecticide Residues in Lettuce. (Compilation; unpublished study received May 26, 1964 under unknown admin. no.; CDL:120061-A.)

00117796. Interregional Research Project No. 4 1968. The Results of Tests on the Amount of Residue Remaining, Including a Description of the Analytical Method Used: Vapona. (Compilation; unpublished study received Apr. 13, 1970 under 0E0875; CDL:091508-A.)

00118084. Shuman Chemical Laboratory, Inc. 1964. Analysis of Vapona Residues: Report No. 1. (Unpublished study received June 17, 1971 under 5011-49; submitted by Carmel Chemical Corp.; CDL:009770-A.)

00118069. Smith, F.; Wheeler, H.; Yeomans, A.; et al. 1964. Dichlorvos Residues on Harvested Tomatoes and Leaf Lettuce Grown in the Greenhouse. (Unpublished study received June 25, 1964 under unknown admin. no.; prepared by U.S. Agricultural Research Service, Fruit & Vegetable Insects Branch and Pesticide Chemicals Research Branch, submitted by Plant Products Corp.; CDL:123044-A.)

Discussion of the data:

Shell Chemical Co. (MRID 00033139) submitted data from two tests conducted in OH concerning residues of DDVP in or on greenhouse-grown lettuce harvested at various intervals after a single application of a 10% aerosol formulation (presumably a PrL) at 0.1 lb ai/50,000 cu. ft (1x) or 0.1 lb ai/100,000 cu. ft (0.5x). Residues in or on one sample harvested 24 hours posttreatment at 0.1 lb ai/50,000 cu. ft (1x) were 0.31 ppm DDVP (0.53 ppm, expressed as naled equivalents.) Samples (two per interval) harvested 8 and 48 hours posttreatment yielded residues of DDVP at 0.41 ppm (0.71 ppm) and 0.28 ppm (0.48 ppm), respectively; values in parentheses reflect residues of DDVP expressed as naled equivalents. Residues in or on lettuce samples (one per interval) harvested 24 and 48 hours after application at 0.1 lb/100,000 cu. ft. (0.5x) were nondetectable (<0.1 ppm). Residues of DDVP in or on one sample harvested 4 hours posttreatment were 4.4 ppm (7.57 ppm expressed

as naled equivalents). Method recovery was 92-102% at fortification levels of 0.05-0.1 ppm. Analysis was accomplished using an adequate enzyme inhibition/spectrophotometric method (MMS-30/60). Residues in control samples were 0.02-0.04 ppm. DDVP residue values were corrected for plant controls prior to calculation of naled equivalents. Samples from the 0.5x treatment regimen were frozen for an unspecified period of time; no information regarding sample storage conditions was provided from the rate test. It was not specified whether these data reflect residues in or on lettuce with or without wrapper leaves.

Summit Chemical Co. (MRID 00119536) submitted data from 14 tests conducted in MD (by USDA) concerning residues of DDVP in or on greenhouse-grown lettuce (leaf and Bibb) harvested 16-160 hours after a single application of a 10% aerosol (presumably a PrL formulation) at 1 g ai/1,000 cu. ft (0.11 lb ai/50,000 cu. ft.; 1.1x). Residues in or on nine samples each of leaf and Bibb lettuce ranged from nondetectable ("0.00" ppm, limit of detection not stated) to 0.026 ppm (0.04 ppm expressed as naled equivalents.) Method recovery was 96% at a fortification level of 0.05 ppm. An adequate analytical method (GC equipped with a flame photometric phosphorus detector) was used for residue analyses. No residue was detected in or on the leaf or Bibb untreated plant controls. Residue values were not corrected for control values or method recovery. Upon harvest, samples were refrigerated for an unspecified period of time.

Fuller System, Inc. (MRID 00118572) submitted data from a single MD test concerning residues of DDVP in or on greenhouse-grown lettuce harvested 16-350 hours after application as a fog (ignited from a fuel canister) of an unspecified 11% formulation released at 1.6 g/1,000 cu. ft (0.18 lb ai/50,000 cu. ft; 1.1x). Residues of DDVP in or on one sample harvested 16 hours after application where 2.35 ppm (4.04 ppm expressed as naled equivalents). By the 88-hour sample interval, residues of DDVP had declined to 0.027 ppm (0.046 ppm expressed as naled equivalents) and were nondetectable ("0.00" ppm; limit of detection unspecified) in or on six lettuce samples harvested 112-350 hours posttreatment. Analyses were performed using a GLC

equipped with a flame photometric detector. No residue was detected on one untreated plant control sample. Recovery efficiency of samples fortified at 0.02-0.5 ppm DDVP was 96% from pulp of "fruit and vegetables" (lettuce, however, was not specifically mentioned). Residue values were not corrected for recovery efficiency and control values.

IR-4 (MRID 00082271) submitted data from a single test conducted in MD concerning residues of DDVP in or on greenhouse-grown lettuce harvested at various intervals following application of a 10% aerosol (presumably a PrL formulation) at 1 g/1,000 cu. ft (0.11 lb ai/50,000 cu. ft; 1.1x). Residues in or on one sample harvested 2 hours after application were 0.24 ppm (0.41 ppm calculated as naled equivalents). Residues in or on one sample each harvested 1, 2, 4 and 8 days after application were nondetectable (<0.01 ppm). Analysis was performed utilizing GLC with flame photometric detection. Recovery was 92-98% following fortification at 0.01 ppm level. It was not stated if residue values were corrected for method recovery or control values and plant control values were not provided. Samples were either analyzed immediately, or frozen for an unspecified period of time prior to analysis.

The available data are insufficient to assess the established 1 ppm tolerance for residues of DDVP (expressed as naled) in or on lettuce because the data reflect single applications, whereas multiple applications are permitted. In addition, none of the studies specified whether sample analysis was performed on lettuce with or without wrapper leaves.

Fruiting Vegetables (except Cucurbits) Group

Conclusions for the Fruiting Vegetables (except Cucurbits) Group:

A crop group tolerance is not appropriate at the present time for the following reasons:

- o Tomato is the only group member for which DDVP formulations are registered for use.

- o Additional data are required to support the established tolerance for DDVP residues in or on tomatoes.

Tomatoes

Tolerance:

A tolerance of 0.5 ppm has been established for residues of DDVP (expressed as naled equivalents) in or on tomatoes following pre- and postharvest application [40 CFR 180.235(a)]. [Note: The 0.5 ppm tolerance was erroneously printed as 0.05 ppm in the 1984 40 CFR. The intended 0.5 ppm tolerance was published in the Federal Register Oct. 20, 1982 p. 46719 (Vol 47, No 203). Also, the tolerance should not be expressed in terms of naled, but as DDVP per se.]

Use directions and limitations:

The 10% RTU formulation is registered for use on greenhouse-grown tomatoes at 16.7 fl. oz product per 50,000 cu. ft; the 10% PrL is registered for use on greenhouse-grown tomatoes at 1 lb of product per 50,000 cu. ft. Liquid ready-to-use formulations should be applied using a thermal fog generator. For spider mites, four treatments may be made at 3-day intervals and this treatment schedule repeated monthly. A 24-hour PHI is in effect.

The 1% D and 4 lb/gal EC formulations are registered for postharvest appli-

cation to tomatoes at 0.01 lb ai/ton of fruit. Application is to be made immediately after harvest, thoroughly covering fruit stored in containers. Additional treatment may be made at the receiving station or cannery yard after transit.

Conclusions:

The available data do not support the established tolerance for residues of DDVP in or on tomatoes, following registered use, for the following reasons: i) no data were submitted reflecting residues following maximum permissible, combined preharvest and postharvest application rates; ii) tolerance-exceeding residues were reported from single postharvest applications at 0.2 and 1x the maximum registered rate (MRIDs 00115993 and 00033144); and iii) insufficient data were submitted regarding the potential for concentration of residues in the processed tomato products. Therefore, the following additional data are required:

- Residues of concern in or on tomatoes following multiple, pre- and postharvest applications at maximum rates. Acceptable examples of such data would be values generated from tomatoes harvested 24 hours after the last of four applications administered at 3-day intervals using either 16.7 fl. oz. of the 10% RTU or 1 lb of the 10% PrL per 50,000 cu. ft. The four-treatment application schedule should be repeated monthly during the growing season prior to the final series. Harvested tomatoes should be treated twice postharvest with the 1% D and, in separate tests, the 4 lb/gal EC formulation at the rate of 0.01 lb ai/ton of fruit. Tomatoes should be treated immediatly after harvest and again after transit and samples collected for analysis immediately following the latter treatment.

- Data must be provided for tomato processed products: wet and dry pomace, puree, catsup, and juice, to determine whether residues concentrate upon processing. Exaggerated rates of application may be necessary to ensure that measurable, weathered residues occur on the raw commodity. If residues are found to concentrate upon processing, then appropriate food/feed additive tolerances must be proposed.

There is no Mexican tolerance for residues of DDVP in or on tomatoes. A Canadian tolerance of 0.25 ppm has been established for residues of DDVP in or on tomatoes, and a Codex MRL of 0.5 ppm has been established for residues of DDVP in or on vegetables (except lettuce).

References (used):

00033144. Shell Chemical Company. 1963. Determination of Vapona Insecticide & Dichloroacetaldehyde Residues in Cucumbers, Lettuce, Mushrooms, Spinach & Tomatoes following Application of Vapona Insecticide: RES-63-116. (Unpublished study received May 26, 1964 under 201-125; CDL:000814-F.)

00107572. Jones, W.; Miles, E.; Hill, K. 1969. Report of Residue Analysis: PCB-69-8. (U.S. Agricultural Research Service, Entomology Research Div., Pesticide Chemicals Research Branch, Analytical Investigations; unpublished study; CDL:022406-A.)

00115993. Shell Chemical Co. 1966. The Results of Tests on the Amount of Residues Remaining, Including a Description of the Analytical Methods Used: Vapona. (Compilation; unpublished study received May 27, 1967 under 7F0623; CDL:090815-A.)

00117686. Shell Chemical Co. 1962. Determination of Residues of Vapona Insecticide in Various Crops. (Compilation; unpublished study received Apr. 2, 1962 under unknown Admin. No.; CDL:120065-A.)

00118169. Smith, F.; Wheeler, H.; Yeomans, A.; et al. 1964. Dichlorvos Residues on Harvested Tomatoes and Leaf Lettuce Grown in the Greenhouse. (Unpublished study received June 25, 1964 under unknown admin. no.; prepared by U.S.A.R.S., Fruit & Vegetable Insects Branch and Pesticide Chemicals Research Branch, submitted by Plant Products Corp.; CDL:123044-A.

00118572. Jones, W.; Miles, E.; Hill, K. 1970. Report of Residue Analysis: Dichlorvos. (Unpublished study received June 18, 1974 under 1327-36; prepared by U.S.A.R.S., Entomology Research Div., Pesticide Chemicals Research Branch, Analytical Investigations, submitted by Fuller System, Inc.; CDL: 024527-A.)

References (not used):

[The following references contain data that are irrelevant, illegible, or previously-cited.]

00117796. Interregional Research Project No. 4. 1968. The Results of Tests on the Amount of Residue Remaining, Including a Description of the Analytical Method Used: Vapona. (Compilation; unpublished study received Apr. 13, 1970 under OE0875; CDL:091508-A.)

00118084. Shuman Chemical Laboratory, Inc. 1964. Analysis of Vapona Residues: Report No. 1. (Unpublished study received June 17, 1971 under 5011-491; submitted by Carmel Chemical Corp; CDL:009770-A.)

00118141. Smith, F. 1963. DDVP Residues--Tomato Fruits and Collard Leaves: Vapona Aerosols. (Unpublished study received May 20, 1963 under 1187-62; submitted by Virginia Chemicals, Inc.; CDL:101579-A.)

00119536. Summit Chemical Co. 1969. Study: DDVP Residue in Tomatoes and Other Selected Crops. (Compilation; unpublished study received Sep. 25,

00141129. Shell Chemical Co. 1962. Evaluation of Residue Data: DDVP on Tomatoes. Unpublished study. 2 p.
1970 under 6218-13; CDL:007833-A.)

Discussion of the data:

Shell Chemical Co. (MRID 00117686) submitted data from two tests conducted in OH concerning residues of DDVP in or on tomatoes harvested 15 minutes, 13 hours, and 1 day after a single preharvest greenhouse application of a 10% aerosol (presumably a PrL formulation) at 20 g ai/14,000 cu. ft or 1 g/1,000 cu. ft [0.157 lb ai/50,000 cu. ft (1.57x) and 0.11 lb ai/50,000 cu. ft (1.1x), respectively.] Residues of DDVP in or on samples treated at the 0.157 lb ai/50,000 cu. ft rate ranged from nondetectable (<0.05 ppm) 1 day after treatment to 0.13 ppm (0.22 ppm naled equivalents) 15 minutes after application. Residues in or on one sample harvested 1 day after treatment were nondetectable. Residues in or on three samples harvested 15 minutes to 2 days after application at the rate of 0.11 lb ai/50,000 cu. ft (1.1x) were nondetectable (<0.1 ppm DDVP). Analyses were performed using an enzyme inhibition/spectrophotometric method (MMS-30/60) considered adequate for data collection. Recovery efficiencies were calculated by fortifying extracts. This procedure is unacceptable because it does not portray residue losses which can occur during the initial extraction. Residues in or on two untreated control samples were 0.01-0.02 ppm. The data do not reflect the maximum number of permissible of preharvested applications. No postharvest treatment data were presented. Samples were stored frozen for an unspecified period of time prior to analysis.

Fuller System, Inc. (MRID 00118572) submitted data from a test conducted in MD by the USDA concerning residues of DDVP in or on tomatoes harvested 16-350 hours after application of an 11% Impr formulation [not specifically registered for greenhouse tomatoes, but similar to thermally-generated vapor from 10% RTU formulation] at 1.69 g/1,000 cu. ft (0.19 lb/50,000 cu. ft; 1.9x RTU rate). Treatment consisted of ignition of the canister which released DDVP vapors into the air. Residues in or on ten samples ranged from 0.074 ppm DDVP (0.127 ppm expressed as naled equivalents) 16 hours after application to "0.00" ppm (no lower limit of detection given) 88-350 hrs after application (7 samples). Residues at 40 and 60 hours posttreatment were 0.012 and 0.0137 ppm DDVP (0.021 and 0.023 ppm naled equivalents). Analyses were performed on whole tomato fruits using a gas/liquid chromatograph

equipped with a flame photometric/phosphorous filter detector. Average recovery of an unspecified number of samples fortified with 0.02-0.05 ppm DDVP was 96%. No residues were detected in or on plant controls. Residue values were not corrected for recovery. Samples were stored in methylene chloride for an unspecified time until analysis. Only one preharvest treatment was applied. No postharvest applications were made.

The USDA (MRID 00107572) conducted a test in MD concerning residues of DDVP in or on greenhouse-grown tomatoes harvested 16-160 hours after application of a 10% PrL ("10% aerosol") at 1 g ai/1,000 cu. ft (11 lb ai/50,000 cu. ft; 1.1x). Residues ranged from "0.00" ppm (no lower limit of detection given) to 0.023 ppm DDVP (0.04 ppm naled equivalents). Residues in or on two samples analyzed 16 hours after application were "0.00" and 0.008 ppm DDVP (0.01 ppm naled equivalents). Residues in or on three tomato samples harvested 40-88 hours after treatment were 0.003-0.023 ppm DDVP (0.01-0.04 ppm naled equivalents). Residues in or on two tomato samples were nondetectable 112 and 160 hours after treatment. Average recovery of an unspecified number of samples fortified at 0.04 ppm DDVP was 97%. Analyses were performed using a gas/liquid chromatograph equipped with a flame photometric detector and a phosphorous-sensitive filter. No lower limit of detection was provided. Residues in or on one untreated control sample were nondetectable ("0.00"). This report did not state whether residues were corrected for recovery efficiency. Samples were stored refrigerated in polyethylene bags for an unspecified period of time prior to analysis.

Shell Chemical Co. (MRID 00115993) submitted data from 13 tests [IN (5), NJ (4), CA (2) and OH (2); number of tests per state appear parenthetically] concerning residues of DDVP in or on tomatoes following postharvest application of 1% D at 0.002, 0.01, and 0.02 lb ai/ton (0.2x, 1x, and 2x, respectively), or 1 tsp/box.

In a study conducted in IN, residues in or on two composite samples of tomatoes analyzed 0 hour after one postharvest application of the 1% D at 0.002 lb ai/ton (0.2x) were 0.35 and 0.36 ppm DDVP (0.55 and 0.57 ppm naled equivalents). Untreated control samples yielded DDVP residues of 0.03 ppm.

In a study conducted in NJ, residues in or on two samples of unwashed tomatoes harvested 0 hour after one postharvest application of the 1% D at 0.01 lb ai/ton (1x) were <0.03 and 0.04 ppm DDVP (<0.05 and 0.07 ppm naled equivalents). Residues in or on two samples of washed fruit from the same regimen were <0.03 ppm (<0.05 ppm naled equivalents). Residues in or on two samples analyzed 0 hour after one application of the 1% D formulation at 0.02 lb ai/ton (2x) were 0.07 and 0.21 ppm DDVP (0.12 and 0.36 ppm naled equivalents). Two samples of washed tomatoes analyzed following identical treatment yielded residues of 0.06 and 0.08 ppm DDVP (0.10 and 0.14 ppm naled equivalents). Residues in or on two samples analyzed 0 hour after two postharvest treatments (at 8-day intervals) of the 1% D at 0.11 lb ai/ton (1x) were <0.03 and 0.07 ppm (<0.05 and 0.12 ppm naled equivalents). Residues in or on two samples of washed fruit from the regimen were <0.03 and 0.10 ppm DDVP (<0.05 and 0.17 ppm naled). Residues in or on two samples harvested 0 hour after two applications (at 8-day intervals) of the same formulation at 0.02 lb ai/ton (2x) were 0.09 and 0.11 ppm DDVP (0.15 and 0.19 ppm naled equivalents) and <0.02 and 0.04 ppm DCA. Residues in or on two samples of washed tomatoes from the same regimen and harvested at 0 hour, were 0.12 and 0.17 ppm DDVP (0.21 and 0.29 ppm naled equivalents).

In another study conducted in IN, residues in or on one sample each of tomatoes treated at 0.01 or 0.02 lb ai/ ton using the 1% D formulation and harvested eight hours after treatment were 0.2 and 0.3 ppm DDVP (0.34 and 0.52 ppm naled equivalents). Residues were corrected for control values; the maximum apparent residue was 0.2 ppm DDVP. Residues of DCA in or on one tomato sample treated at 0.01 lb ai/ton were <0.05 ppm. Data were collected using a human blood-enzyme inhibition/spectrophotometric method (MMS-30/64) and GLC method PMS-G-900/66. Both methods are acceptable for data collection. Stated detection limits ranged from 0.02-0.04 ppm DDVP.

Data also were submitted (MRID 00115993) for processed tomato products including juice and canned tomatoes. Juice was processed from tomatoes bearing nondetectable residues (<0.01 ppm DDVP) in or on seven samples, or from tomatoes on which no residue data were gathered (an eighth sample). Residues of DDVP in two juice samples were nondetectable (<0.02 or <0.01

ppm; detection limit varied with test), and were 0.1 ppm in two additional juice samples. One sample of canned tomatoes contained <0.04 ppm DDVP residues processed from fruit bearing 0.05 ppm DDVP (0.09 ppm naled equivalents.) Analyses were performed for DDVP residues using an enzyme-inhibition/spectrophotometric method (MMS-30/64). All samples were frozen for an unspecified period until analyzed.

Shell Chemical Co (MRID 00033144) submitted data from a test in CA concerning residues of DDVP in or on tomatoes following one postharvest application of technical DDVP (not a registered formulation) at approximately 5 ppm (0.01 lb ai/ton; 1x). Residues, corrected for apparent residues on plant controls, ranged from 1.3 to 5 ppm DDVP (2.2 to 8.6 ppm naled equivalents). Residues 1 hour after application were 5 ppm DDVP (8.6 ppm naled equivalents), and 0.09 ppm DCA in or on two samples. Residues in or on two samples harvested 7 days after application were 1.3 and 1.4 ppm DDVP (2.2 and 2.4 ppm naled equivalents), and 0.10 and 0.11 ppm DCA. An enzyme-inhibition/spectrophotometric method, MMS-30/60, was used for analysis of DDVP. Recovery efficiency for samples fortified at 0.1 ppm were 65-100%. DCA was analyzed by gas/liquid chromatography, using an electron capture detector (method MMS-50/63). Recovery efficiency for samples fortified with 0.1 ppm DCA were 66-92%. Residue values were corrected for apparent residues contained in plant controls; these values ranged from 0.01 to 0.07 ppm DDVP (0.02 to 0.12 ppm naled equivalents) and <0.01 to 0.02 ppm DCA. Samples were stored at approximately 2 C until extracted.

Residue data generated by the USDA (MRID 00118169) were submitted from ten tests conducted in MD concerning residues of DDVP in or on tomatoes harvested 0 hour to 10 days after 1 hour to 1 day exposure to a 10% aerosol, mist, or smoke. Residues ranged from nondetectable ("0", no lower limit of detection given) to 10.6 ppm DDVP (18.2 ppm naled equivalents) from smoke application, 0 to 8.7 ppm DDVP (15.0 ppm naled equivalents) from the mist application, and 0 to 11.9 ppm DDVP (20.5 ppm naled equivalents) from the aerosol application. Since rates for preharvest treatment are not expressed in terms of hours of exposure, these data are not useful for tolerance evaluation. However, the data are summarized here for informational purposes.

Residues in or on three samples harvested 0 hour, 1 day, or 3 days after 10 hours of exposure to DDVP smoke were 10.6, 3.1, and 1.8 ppm DDVP (18.2, 5.3, and 3.1 ppm naled equivalents), respectively. No residues were detected at 7- and 10-day postharvest intervals. Residues in or on two samples harvested 0 hour and 1 day after 3 hours of exposure to the smoke were 0.8 and 0.25 ppm DDVP, respectively (1.4 and 0.43 ppm naled equivalents). No residues were detected at 3 or 7 days. Residues in or on three samples harvested 0 hour, 1 day, or 3 days after 1 day of exposure to DDVP smoke were 0.2, 0.2 and 0.45 ppm DDVP (0.3, 0.3 and 0.77 ppm naled equivalents). Residues in or on two samples harvested 0.3 hour or 1 day after 12 hours of exposure to the mist were 0.3 and 5.9 ppm DDVP (0.5 and 10 ppm naled equivalents). Residues in or on three samples harvested 0 hour, 1 day, and 2 days after 10 hours of exposure to the mist were 0.1, 8.7 and 3.3 ppm DDVP (0.2, 15.0 and 5.7 ppm naled equivalents), respectively. Residues in or on four samples harvested 0 hour or 1 day after 1 hour of exposure to the aerosol were 0.7 and 0.8 ppm DDVP (1.2 and 1.4 ppm naled equivalents), and 0.0 and 0.2 ppm DDVP, respectively (0.0 and 0.3 ppm naled equivalents). Residues in or on four samples harvested 0 hour, 1 day, or 2 days after exposure for 8 hours were 1.8, 0.1 and 0.2 ppm DDVP (3.1, 0.2, and 0.3 ppm naled). Residues in or on two samples harvested 0 hour or 1 day after 10 hours of exposure to the 10% aerosol were 0.2 and 0.3 ppm DDVP respectively (0.3 and 0.5 ppm naled equivalents). Residues in or on two samples harvested 0 hour after 1 day of exposure to the aerosol were 9.1 and 11.9 ppm DDVP (15.7 and 20.5 ppm naled equivalents). Residues in or on two samples harvested 1 day after exposure for 1 day were 3.9 and 8.3 ppm DDVP (6.7 and 14.2 ppm naled equivalents). Residues in or on two samples harvested 3 days after exposure for 1 day were 0.4 and 3.3 ppm DDVP (0.7 and 5.7 ppm naled equivalents). Residues in or on four samples harvested 0 hour, 1 day, 3 days, and 7 days after 2 hours exposure to the aerosol were 0.2, 0.4, 0.1, and 0.1 ppm, respectively; (0.3, 0.4, 0.2, and 0.2 ppm naled equivalents, respectively.)

Cucurbit Vegetables Group

Conclusions for the Cucurbit Vegetables Group:

A crop group tolerance is not appropriate at the present time because DDVP formulations are registered for use only on cucumbers.

Cucumber

Tolerance:

A tolerance of 0.5 ppm has been established for residues of DDVP (expressed as naled) in or on cucumbers [40 CFR 180.235(a)]. [Note: The tolerance should not be expressed in terms of naled, but as DDVP per se.]

Use directions and limitations:

The 10% RTU formulation is registered for fumigation (fogging applications) use on greenhouse-grown cucumbers at 16.7 fl. oz. of product per 50,000 cu. ft, using a thermal fog generator. The 10% PrL is registered for fumigation use on greenhouse-grown cucumbers at 1 lb of product (0.1 lb ai) per 50,000 cu. ft. Four applications may be made at 3-day intervals, and the entire schedule may be repeated monthly. A 24-hour PHI is in effect.

Conclusions

The available data are insufficient to assess the established tolerance for residues of DDVP in or on cucumbers because the maximum permissible number of applications was not reflected in the data. The following data are required:

- Residues of concern in or on cucumbers harvested 24 hours after the final application in a treatment regimen consisting of four fogging treatments at 3-day intervals repeated monthly using either the 10% RTU formulation at 16.7 fl. oz. product per 50,000 cu. ft or the 10% PrL at 1 lb product (0.1 lb ai) per 50,000 cu. ft. Application of the RTU should be made using a thermal fog generator.

No Canadian or Mexican tolerance has been established. A Codex MRL of 0.5 ppm has been established for residues of DDVP in or on vegetables (except lettuce).

References (used):

00082271. Interregional Research Project Number 4. 1968. Residue Study of Dichlorovos on Tomatoes, Lettuce and Cucumbers. (Compilation; unpublished study received on unknown date under OE0875; CDL:097531-B.)

00107572. Jones, W.; Miles, E.; Hill, K. 1969. Report of Residue Analysis: PCB-69-8. (U.S. Agricultural Research Service, Entomology Research Div., Pesticide Chemicals Research Branch, Analytical Investigations; unpublished study; CDL:022406-A.)

00118572. Jones, W.; Miles, E.; Hill, K. 1970. Report of Residue Analysis: Dichlorvos. (Unpublished study received June 18, 1974 under 1327-36; prepared by U.S. Agricultural Research Service, Entomology Research Div., Pesticide Chemicals Research Branch, Analytical Investigations, submitted by Fuller System, Inc.; CDL:024527-A.)

References (not used):

[The following references are illegible, duplicate previously-cited references or lack sufficient detail for evaluation.]

00033144. Shell Chemical Company. 1963. Determination of Vapona Insecticide & Dichloroacetaldehyde Residues in Cucumbers, Lettuce, Mushrooms, Spinach & Tomatoes following Application of Vapona Insecticide: RES-63-116. (Unpublished study received May 26, 1964 under 201-125; CDL:000814-F.)

00117686. Shell Chemical Co. 1962. Determination of Residues of Vapona Insecticide in Various Crops. (Compilation; unpublished study received Apr.2, 1962 under unknown admin. no.; CDL:120065-A.)

00117796. Interregional Research Project No. 4. 1968. The Results of Tests on the Amount of Residue Remaining, Including a Description of the Analytical Method Used: Vapona. (Compilation; unpublished study received Apr. 13, 1970 under 0E0875; CDL:091508-A.)

00119536. Summit Chemical Co. 1969. Study: DDVP Residue in Tomatoes and Other Selected Crops. (Compilation; unpublished study received Sep. 25, 1970 under 6218-13; CDL:007833-A.)

Discussion of the data:

Fuller System, Inc. (MRID 00118572) submitted data from a single test conducted in MD concerning residues of DDVP in or on greenhouse-grown cucumbers harvested 16-350 hours after application (as a fog ignited from a fuel canister) of an unspecified 11% formulation released at 1.6 g/1,000 cu. ft (0.18 lb ai/50,000 cu. ft; 1.1x). Residues of DDVP in or on cucumbers (one sample per harvest interval) were 0.198 ppm (0.34 ppm), 0.019 ppm (0.03 ppm), and 0.036 ppm (0.06 ppm) at 16, 40, and 64 hours posttreatment, respectively; values in parentheses reflect residues of DDVP expressed as naled equivalents. Residues of DDVP were nondetectable ("0.00" ppm, limit of detection not quantified) in or on seven cucumber samples harvested 88-350 hours posttreatment. Residue analysis was accomplished using an adequate method (GLC equipped with a flame photometric detector). Residues in or on one untreated control sample were nondetectable. Recovery efficiency was 96% from pulp of "fruit and vegetables" (cucumber was not specifically mentioned) fortified with DDVP at 0.02-0.5 ppm.

IR-4 (MRID 00082271) submitted data from a single test conducted in MD concerning residues of DDVP in or on greenhouse-grown cucumbers harvested 2 hours to 8 days following application of a 10% aerosol (presumably a PrL formulation) at 1 g/ 1,000 cu. ft (0.11 lb ai/50,000 cu. ft; 1.1x). Residues of DDVP in or on one cucumber sample harvested 2 hours after application were 0.01 ppm (0.017 ppm expressed as naled equivalents). Residues in or on one sample each harvested 1, 2, 4 and 8 days posttreatment were nondetectable (<0.01 ppm). Analysis was performed utilizing gas/liquid

chromatography with a flame photometric detector. Recovery was 92-98% from samples fortified at 0.01 ppm. It was not stated if residue values were corrected. No untreated plant control values were provided. Samples were either analyzed immediately, or frozen for an unspecified period of time.

The USDA (MRID 00107572) conducted a single trial in MD concerning residues of DDVP in or on greenhouse-grown cucumbers harvested 16 to 400 hours after one application of a 10% aerosol (presumably a PrL formulation) released at 1 g/1,000 cu. ft [0.11 lb ai/50,000 cu. ft; 1.1x]. Surface residues of DDVP (stripped with methylene chloride) on one sample each of cucumber fruit harvested at various intervals from 16 to 40 hours posttreatment were nondetectable ("0.000" ppm; limit of detection not specified) to 0.004 ppm (0.007 ppm naled equivalents). Residues in or on cucumbers macerated after surface stripping and harvested at the same posttreatment intervals were nondetectable ("0.000" ppm; limit of detection not specified) to 0.055 ppm (0.096 ppm naled equivalents). Analyses were performed by GLC equipped with a flame photometric detector. Cucumber fruits were first stripped in CH_2Cl_2, then subjected to extraction. [No data were presented to indicate if the stripped residue correlated with the specific samples from which extracted residue was obtained]. Recovery was 98% for pulp, and 95% from foliage. Residues in pulp from untreated controls were nondetectable ("0.00" ppm; limit of detection not specified) and were <0.002 ppm in surface stripped whole fruit. Samples were refrigerated for an unspecified period of time prior to analysis.

Miscellaneous Commodities

Figs

Tolerances:

A tolerance of 0.1 ppm has been established for residues of DDVP in or on fresh figs [40 CFR 180.235(a)]. A tolerance of 0.5 ppm has been established for the same residues in or on dried figs [21 CFR 193.140].

Use directions and limitations:

The 2 lb/gal EC formulation is proposed for use on figs at 2 lb ai/300-400 gal/A. Full coverage sprays are to be made at the beginning of harvest and continued at 4- to 5-day intervals. A 5-day PHI is proposed. No more than five applications are to be permitted and grazing is to be restricted in treated orchards (revised section B, dated 3/2/82, in PP#1E2510/FAP#1H5309).

Conclusions:

The available data support the established tolerances for residues of DDVP in or on fresh and dried figs provided the proposed use directions are followed. No additional data are required.

No Codex MRL or Mexican or Canadian tolerance has been established for residues of DDVP in or on figs.

Reference (used):

00076809. Shell Chemical Company. 1980. Summary of Residue Data for Dichlorvos in or on Figs. (Compilation; unpublished study, including published data, received May 8, 1981 under 201-125; CDL:070073-A.) [Note: Author designation is incorrect. Data were submitted by IR-4 under PP#1E2510/FAP#1H5309.]

Reference (not used):

[The following MRID duplicates previously cited information.]

00076811. Shell Chemical Company. 1978. Dichlorvos--Dried Figs. (Compilation; unpublished study received May 8, 1981 under 201-125; CDL: 070073-C.)

Discussion of the data:

IR-4 (MRID 00076809 - Shell Chemical Co. erroneously listed as author) submitted data from five tests conducted in CA concerning residues of DDVP in or on fresh and dried figs. In one set of tests, fig trees received multiple foliar spray applications at 5day intervals of the 2 lb/gal EC formulation at 2 or 4 lb ai/A. Samples were taken 5 days after the last of three, four, and five applications. Residues in or on samples of fresh figs, figs air-dried on the ground, and dehydrated figs (48.9 C for 48 hours) are summarized in Table 6; each residue range represents three samples.

Table 6. Residues of DDVP in or on figs (fresh, air-dried, and oven-dehydrated) following treatment with the 2 lb/gal EC formulation.

Treatment rate	Applications	Fresh	Air-dried	Dehydrated
2 lb ai/A (1x)	3	<0.005-0.005	0.048-0.097	---
	4	0.008-0.020	0.020-0.059	---
	5	<0.005	0.037-0.244	---
4 lb ai/A (2x)	3	0.008-0.010	0.144-0.297	0.012-0.025
	4	0.013-0.050	0.116-0.363	<0.005-0.027
	5	<0.005	0.197-0.527	0.021-0.054

In another test, trees were treated with a single application of the 2 lb/gal EC formulation at 2 lb ai/A (1x). Residues of DDVP in or on two samples of fresh figs at 0 hour and 1 day after application were 0.72 and 0.19 ppm, respectively. Residues of DDVP were 0.08 and 0.07 ppm at two and four days, respectively. Residues were nondetectable at 6 and 10 days.

In another test, figs were treated with a single application of the EC formulation at the rate of 2 lb ai/A (1x). Samples of figs were harvested immediately after and 10 days after treatment, washed, dried at 48.9 C for 48 hr., and frozen at an unspecified temperature until analyzed. Residues were 0.12, 0.02, 0.04, 0.03, 0.01 and 0.02 ppm at 0 hour and 1, 3, 5, 7,

and 10 days posttreatment, respectively. Residues on control samples were not detectable.

In a fourth test, four applications were made at 5-6 day intervals at either 2 or 4 lb ai/A. Single samples were taken after each application at both rates. Residues of DDVP in or on eight samples were 0.01-0.05 ppm. No trends suggesting increasing residue concentrations following multiple applications were observed.

In a final test, samples of ground-dried figs were collected and stored frozen following one, three or five applications of the 2 lb ai/gal EC formulation at the rate of 2 or 4 lb/A. Following a single application, values of 0.66 and 1.02 ppm (2 and 4 lb ai/A, respectively) were observed at 0 hour after treatment. Residues on figs collected 24-72 hours after treatment bore residues ranging from nondetectable to 0.30 ppm. Samples collected after the third application (applications were administered at 5-day intervals) bore residues of 0.73 and 1.38 ppm (2 and 4 lb ai/A, respectively) at 0 hour after treatment. Residues on samples collected 24-72 hours after treatment exhibited residues ranging from nondetectable to 0.28 ppm. Following treatment with five applications at 4-5 day intervals, residues were 2.04, 0.85, and 0.98 ppm at 0, 24 and 48 hours, respectively, at the 2 lb ai/A rate, and 10.69, 3.03 and 0.85 ppm at identical sampling intervals following application at the rate of 4 lb ai/A.

Recovery efficiencies of samples of figs fortified at an unspecified level were 80-88%. An adequate GLC method, RM-3-G-3, was used for residue determination. Geographic representation was adequate because virtually all of the domestically-grown figs are produced in CA (Agricultural Statistics, 1984, pp 209, 231).

Mushrooms

Tolerance:

A tolerance of 0.5 ppm has been established for residues of DDVP (expressed as naled) in or on mushrooms [40 CFR 180.235(a)]. [Note: The tolerance should not be expressed as naled, but as DDVP per se.]

Use directions and limitations:

The 2 lb/gal EC formulation is registered for use in and on the outside of mushroom houses at 0.5-1 pt ai/100 sq. ft, applied as a 0.5% finished spray. Applications may be made to inside walls, door, ventilators and cracks with a brush or a coarse, wet spray. Inside walls may not be sprayed after mushrooms appear above soil beds. After mushrooms appear, treatment is limited to outside walls. A 10 lb/gal EC is also registered for use in mushroom houses at 100 g ai/ 50,000 cu. ft applied as an aerosol, or vapor by a hot pipe method. Application may be made every 4 days as a preventative measure, or when infestations are present and may be repeated as needed. The 10% RTU is registered for use in mushroom houses at 33-50 fl. oz. of product per 50,000 cu. ft as a thermally generated application. Fog must be directed downward from the center aisle of the room. Direct nozzle away from beds, and do not allow the end of the gun to be within 10 ft of mushrooms. Use twice weekly during spawn run and thereafter, as needed. The 50% SC/L MAI (formulated with 1,1,1-trichloroethane) is registered similarly at 1 pt of product per 4.4 gal of solvent or 50 g ai/50,000 cu. ft. A 1-day PHI is in effect.

Conclusions:

The available data support the established tolerance for residues of DDVP in or on mushrooms. No additional data are required.

No Canadian or Mexican tolerance has been established for residues of DDVP in or on mushrooms. A Codex MRL of 0.5 ppm has been established for residues of DDVP in or on mushrooms.

References (used):

00074658. Interregional Research Project Number 4. 1970. Residue Data on Vapona in Mushrooms. (Reports by various sources; unpublished study received on unknown date under 1E1100; CDL:090860-A.)

00117686. Shell Chemical Co. 1962. Determination of Residues of Vapona Insecticide in Various Crops. (Compilation; unpublished study received Apr. 2, 1962 under unknown admin. no.; CDL:120065-A.)

00117690. Shell Chemical Co. 1963. Determination of Vapona Insecticide Residues in Mushrooms, Banana Peel and Pulp. (Compilation; unpublished study received May 26, 1964 under 201-136; CDL:120071-A.)

References (not used):

[The following references contain irrelevant or previously cited data/information.]

00033142. Shell Chemical Company. 1962. Determination of Vapona Insecticide Residues in Mushrooms Following Application of This Toxicant: RES-62-30. (Unpublished study received May 26, 1964 under 201-125; CDL:000814-D.)

00033144. Shell Chemical Company. 1963. Determination of Vapona Insecticide & Dichloroacetaldehyde Residues in Cucumbers, Lettuce, Mushrooms, Spinach & Tomatoes Following Application of Vapona Insecticide: RES-63-116. (Unpublished study received May 26, 1964 under 201-125; CDL:000814-F.)

Discussion of the data:

Shell Chemical Co. (MRID 00117686) submitted data from six tests [PA(5), CA(1)] concerning residues of DDVP in or on mushrooms harvested 24-120 hours following one to three applications of an unspecified formulation as a hot plate-induced vapor at 4 g/1,000 cu. ft (200 g ai/50,000 cu. ft; 2x the 10 lb/gal EC rate) or one to four applications of aerosol at 0.9 to 6.0 g/1,000 cu. ft (60 to 300 g ai/50,000 cu. ft; 0.6 to 3x the 10 lb/gal EC rate). Residues were nondetectable (<0.1 ppm) in or on all 32 samples.

Analysis was performed by an enzyme inhibition/spectrophotometric method, MMS-30/60, which is acceptable for data collection. Recovery efficiencies were not determined properly because efficiencies were calculated based on fortification of mushroom extracts rather than original crop samples. Furthermore, extracts were fortified at levels below the stated detection limit. Residue values were corrected for apparent residues in or on plant controls, which ranged from nondetectable (<0.1 ppm DDVP; <0.17 ppm naled equivalents) to 0.02 ppm DDVP (0.034 ppm naled equivalents) for three samples.

IR-4 (MRID 00074658) submitted data from 14 tests [DE(8), IL(6), number of tests per state appear parenthetically] concerning residues of DDVP in or on mushrooms harvested 1 to 8 days after three to 12 applications of an unspecified "Vapona" formulation at 2 or 4 g ai/1,000 cu. ft [100 to 200

g ai/50,000 cu. ft; 1x to 2x the 10 lb/gal EC rate; approximately 0.7 to 1.4x the 10% RTU rate]. Residues ranged from nondetectable (<0.005 ppm) to 0.12 ppm DDVP (0.20 ppm naled equivalents). Residues in or on one sample each of mushrooms harvested 1 day after the last of three, five, or six applications of an unspecified formulation at 2 g ai/ 1,000 cu. ft [100 g ai/50,000 cu. ft; 1x the 10 lb/gal EC rate approximately 0.7x the 10% RTU rate] were 0.05, 0.02, and 0.02 ppm DDVP, respectively (0.09, 0.03, and 0.03 ppm naled equivalents). Residues in or on one sample each harvested at the same intervals, but treated at 4 g ai/1,000 cu. ft (1200 g ai/50,000 cu. ft; 2x the 10 lb/gal EC rate; approximately 1.4x the 10% RTU rate) were 0.01, 0.12, and 0.08 ppm DDVP (0.017, 0.20, and 0.136 ppm naled equivalents).

In another series of tests, six or twelve fog applications were made with an unspecified formulation at the rate of 2 g ai/1,000 cu. ft at varying frequencies ranging from 1 to 5 days between applications. Residues in or on four samples harvested 1 day after the final application in both the 6- and 12-application series were nondetectable to 0.03 ppm DDVP. Residues of DDVP on samples collected from 2 to 8 days after the final treatment all were below the stated instrumental detection limit (<0.005 ppm). In another test utilizing "Vapona" (formulation unspecified), samples were harvested 24 hours after the last of three, five, or six applications at 2 or 4 g ai/1,000 cu. ft (100 or 200 g ai/50,000 cu. ft; 1x or 2x the 10% EC rate; 0.7 or 1.4x RTU rate). Residues in or on the samples were $\leq$0.05 ppm DDVP ($\leq$0.08 ppm naled equivalents). Residues were determined by GLC method PMS-G-913/68; method sensitivities were 0.02 or 0.005 ppm depending on the laboratory performing the analyses. Recovery efficiencies, when reported, were 90 and 100%. Samples were frozen for an unspecified period prior to

analysis. Residue values were not corrected for recovery efficiency or apparent residues borne by controls.

Shell Chemical Co. [MRID 00117690] submitted data from one test in PA concerning residues of DDVP in or on mushrooms harvested 0-48 hours following one application of the 4% DDVP-oil formulation at 2 oz. of product per 1,000 cu. ft (approximately 112g/ 50,000 cu. ft; approximately 0.8x RTU rate), applied with a "dyna fog" apparatus. Residues in or on all samples were nondetectable (no lower limit of detection provided).

Residues were determined using an enzyme inhibition/spectrophotometric method (MMS-30/60) which is acceptable for data collection. Recovery efficiencies were improperly determined because DDVP was added to extracts rather than to the mushrooms. Residues were corrected for plant control values, which were 0.01 ppm DDVP in or on two samples (0.017 ppm naled equivalents). Samples were frozen for an unspecified period of time until analyzed.

Additional residue data were generated using various continuous release formulations, none of which resulted in detectable residues in or on mushrooms. Other tests utilizing a hand duster application of a DDVP solution and fog generation of a D formulation were reported. Although residues from these tests were measurable and tolerance-exceeding, they cannot be used to evaluate the tolerance. Use of these formulations is not registered and/or the rate of application cannot be accurately measured.

MAGNITUDE OF THE RESIDUE IN NONPERISHABLE RAW AGRICULTURAL COMMODITIES AND PROCESSED FOOD

Bulk Stored Raw Commodities

Tolerance:

A tolerance of 0.5 ppm has been established for residues of DDVP in or on bulk stored nonperishable raw agricultural commodities regardless of fat content [40 CFR 180.235(a)].

Use directions and limitations:

The 20% impregnated material is registered for use in bulk storage facilities at the rate of one strip per 1,000 cu. ft. of space over the commodity. Suspend strips before moth adults emerge in early spring. Replace after four months.

The 5% SC/L is registered for ULV or fogging application at 50-100 g ai/50,000 cu. ft. Use is limited to professional pest control operators. Remove or cover exposed commodities before spraying. Apply only when building is unoccupied and processing plant is not in operation. Repeat treatment at weekly intervals during season of peak pest activity.

The 2 and 4 lb/gal EC, and 20% EC and PrL formulations are registered for application by ultra low volume equipment at 25-28 g ai/50,000 cu. ft. to bulk stored peanuts in warehouses. Use is limited to professional pest control operators. Treated warehouses should not be entered for a period of 4 hours after treatment or until pesticides have settled.

Conclusions:

The data indicate that the established tolerance will cover residues of DDVP in or on bulk stored nonperishable raw agricultural commodities, regardless of fat content, resulting from continuous exposure to DDVP from impregnated strips. However, no data were submitted reflecting residues in or on

bulk stored commodities resulting from ULV or fogging treatment. With the exception of peanuts, we believe no data are required since exposed commodities must be removed or covered prior to treatment. Data are required, however, for bulk stored peanuts. The following must be submitted:

- o Data reflecting ultra low volume application of DDVP vapors from an EC or PrL formulation at 28 g ai/50,000 cu. ft. to exposed bulk stored peanuts. Samples must be removed for residue analysis 4 hours after application. Alternatively, the registrant may propose a label amendment requiring that exposed peanuts be covered or removed prior to treatment.

Reference (used):

00117747. Shell Chemical Co. 1970. The Results of Tests on the Amount of Residues Remaining, Including a Description of the Analytical Methods Used: Vapona Insecticide. (Compilation; unpublished study received 1970 under 1F1132; CDL:090909-B.)

Discussion of the data:

Shell Chemical Co. (MRID 00117747) submitted data from 94 tests concerning residues in or on bulk stored cocoa beans (6), flour (1), grain sorghum (22), peanuts (2), shelled corn (32), soybeans (21), and wheat (10) (number of tests per commodity given in parentheses) exposed for periods of up to 133 days to DDVP from impregnated strips at rates of 1 strip/1,000 cu. ft. to two strips/28.3 cu. ft. (1x to 70.7x the maximum registered rate). In most tests, the commodity was left undisturbed throughout the exposure period except for the removal of samples. In the single test on flour,

fresh flour was added to the top of the storage bin each week and flour was removed continually from the bottom. This resulted in the removal of approximately 80% of the flour each week, and replenishment with an equal volume weekly. Residues in or on eight weekly samples of flour exposed to 1 strip/600 cu. ft. (1.7x the maximum registered rate) were 0.01-0.03 ppm. Residues in or on 11 samples of husks, kernels, milled husks and kernels, roasted kernels, whole beans, and raw beans of cocoa exposed to 1 strip/ 1,000 cu. ft. (1x) for 55 days were 0.01-0.04 ppm in one test conducted in the Netherlands. Residues in 30 samples of mature cocoa beans exposed to 1 strip/1,000 cu. ft. (1x) for 38-133 days ranged from nondetectable (<0.02 or <0.05 ppm, depending on the test) to 0.11 ppm in two tests conducted in DE.

Bulk stored, farmers stock peanuts and shelled peanuts were exposed to 1 or 2 strips/28.3 cu. ft. (35x to 71x) for up to 158 days. Residues in or on 10 samples of shelled peanuts collected at intervals from 56-158 days from the surface layer of the storage bin were nondetectable (<0.5 ppm). Residues in 12 samples of farmers stock peanuts collected from the surface layer ranged from nondetectable (<0.5 ppm) to 0.7 ppm, and in six samples collected from 4 inches below the surface were nondetectable (<0.5 ppm).

Residues in or on 331 samples of shelled corn exposed to 1 to 2 strips/1,000 cu. ft. (1x to 2x) for 3-133 days ranged from nondetectable (<0.02 ppm or <0.1 ppm, depending on the test) to 0.73 ppm. Residues in or on 128 samples of sorghum exposed to 1 to 2 strips/1,000 cu. ft. (1x to 2x) for 3-119 days ranged from nondetectable (<0.1 ppm) to 0.3 ppm. Residues in or on 118 samples of soybeans exposed to 1 to 2 strips/1,000 cu. ft. (1x to 2x) for 1-126 days ranged from nondetectable (<0.02 to <0.1 ppm, depending on the test) to 0.38 ppm. Finally, residues in or on 88 samples of wheat exposed to 1 strip/1,000 cu. ft. to 1 strip/23 cu. ft. (1x to 43.5x) for 3-126 days ranged from nondetectable (<0.02 ppm or <0.1 ppm, depending on the test) to 0.3 ppm.

Detectable residues rarely were present below the surface 0-2 inches of any commodity, and none of the residues detected in samples taken from two or more inches below the surface were above tolerance. Residues showed a

slight tendency to increase during the first five weeks of exposure and to remain relatively constant or to decline in the following weeks.

Residues were analyzed by Δ pH, colorimetric, or GLC analytical methods: Δ pH method WAMS 30-1 for cocoa beans and flour; colorimetric methods MMS-30/64 for cocoa beans, and Pr 5e-62 for shelled corn, sorghum, soybeans and wheat; GLC method PMS-G-913/69 for soybeans. Recovery from two samples of cocoa beans fortified at 0.05 and 0.1 ppm DDVP and analyzed with method WAMS 30-1 was 76 and 81%, respectively. Recovery from two samples of flour fortified at 0.1 ppm and analyzed by the same method was 110 and 120%. Recovery from five samples of cocoa beans fortified at 0.1 to 0.2 ppm ranged from 70 to 90%. Recovery from two samples each of shelled corn, grain sorghum, and soybeans fortified at 0.1 and 1.0 ppm and analyzed by method Pr 5e-62 ranged from 94 to 104%. Recovery from 18 samples of shelled corn fortified at 0.1-0.2 ppm and analyzed by GLC method PMS-G-913/69 ranged from 60 to 120%.

These data indicate that residues of DDVP on bulk stored commodities, regardless of fat content, continuously exposed to DDVP from impregnated strips will not exceed the established tolerance. We believe the occurrence of tolerance-exceeding residues in or on samples taken from the upper two inches of a commodity stored in large bins to be of little consequence. FDA sampling procedures for bulk stored commodities are to take 10 2 lb samples using probes that extend 1-2 feet into the commodity. These samples are composited and residues are determined in or on subsamples of the 20 lb composite sample (personal communication, Ellis Gunderson, FDA, 13 January 1986).

No data were submitted concerning residues in or on bulk stored commodities resulting from treatment with DDVP by ULV or fogging applicators. With the exception of peanuts, all exposed commodities must be removed or covered prior to treatment. However, ULV applications to stored peanuts may be made without prior removal or cover of the commodity. Therefore, we believe data reflecting this use must be submitted, or the registrant must propose a label amendment requiring the covering or removal of exposed peanuts prior to application or prohibiting the use of DDVP on stored peanuts.

Packaged or Bagged Raw Commodities and Processed Food

Tolerances:

A tolerance of 0.5 ppm has been established for residues of DDVP in or on packaged or bagged nonperishable raw agricultural commodities containing 6 percent fat or less, and a tolerance of 2 ppm has been established for residues of DDVP in or on packaged or bagged nonperishable raw agricultural commodities containing more than 6 percent fat [40 CFR 180.235(a)]. A food additive tolerance of 0.5 ppm has been established for residues of DDVP in packaged or bagged nonperishable processed food (21 CFR 193.140).

Use directions and limitations:

The 20% G may be applied by vapor dispensing machine in warehouses at a rate of 50-100 g ai/50,000 cu. ft. Application period is six hours.

The 5% EC, 5% SC/L, and 5% RTU may be applied by ultra-low-volume applicators at 50-100 g ai/50,000 cu. ft. in warehouses, railroad cars, stationary trucks, flour mills, grain mills, and rice mills. Wait six hours after completion of treatment before ventilating.

The 0.5 SC/L and 0.5% RTU may be applied by ultra-low-volume applicator at 1 fl. oz./1,000 cu. ft. in food warehouses, storage rooms, and similiar areas. Direct spray into corners, under pallets, and around stocks of packaged goods. Allow room to remain closed for 1 hr. after the application.

The 2.95% PrL at 50 g ai/50,000 cu. ft., and the 7% PrL at 60 oz. formulation/ 50,000 cu. ft. may be applied as aerosols in warehouses. Clean out debris and shut off air-conditioners and fans prior to treatment, and ventilate after treatment is completed. The 20% PrL may be applied as an aerosol at 25 g ai/50,000 cu. ft.

The above-noted formulations are for use only by professional pest control operators in warehouses containing packaged and bagged nonperishable raw

agricultural commodities during hours when buildings are unoccupied. Food should be removed and covered, and all food processing surfaces must be covered during treatment or thoroughly cleaned before using. The building should be opened and aired prior to reentry of unprotected workers, or should remain unoccupied by unprotected workers for at least 6 hours after treatment.

Conclusions:

The data indicate that the established tolerances will cover residues of DDVP in or on packaged or bagged nonperishable raw agricultural commodities and processed food resulting from the application of DDVP vapors. However, the available data indicate that residues following use of aerosol treatments will result in tolerance-exceeding residues. The maximum residues likely to occur cannot be estimated because tests were not conducted at or near maximum use rates. Thus, the following data are required.

- o Data depicting residues in packaged or bagged nonperishable processed food (cereals, flour, sugar, cookies, and crackers), and packaged or bagged raw agricultural commodities (dried beans, cocoa beans, coffee beans, corn, nuts, peanuts, and soybeans) collected 6 hours after aerosol treatment with a PrL formulation at 50 g ai/50,000 ft^3 of chamber. An appropriate tolerance revision must be proposed. Alternatively, the registered aerosol uses may be cancelled.

References (used):

00056593. Gillenwater, H.B. 1964. Residues on Loose and Packaged Commodities after Exposure to Dichlorvos (DDVP) Aerosol: Project MQ 1-24:3. (Unpublished study received Apr. 16, 1964 under 5H1748; submitted by Shell Chemical Co.; CDL:221615-Y.)

00056595. U.S. Agricultural Marketing Service. 1964. Dichlorvos (DDVP) Residues on Packaged Subsistence Items Exposed to Vapor Applications in a

126,000 cu. ft. Warehouse Bay. (Market Quality Research Div., Stored-Product Insects Branch, unpublished study; CDL:221615-AA.)

00056596. U.S. Agricultural Marketing Service, Stored-Product Insects Branch. 1963. DDVP Residues on Packaged Commodities after Exposure to a Relatively Constant Vapor Concentration Applied with DDVP-Impregnated Granules: Project BS 1-51:62. (Unpublished study; CDL:221615-A.)

References (not used):

[The following references contain no pertinent residue data.]

00049964. Shell Chemical Company. 1967. Abstract of Petition Proposing Tolerances for Dichlorvos (Vapona (R) Insecticide) on Stored Food Commodities. (Unpublished study received Apr. 4, 1967 under 7H2166; CDL:221633-A.)

00056594. U.S. Agricultural Marketing Service. 1964. Studies on DDVP Vapor and Aerosol Applications in Warehouses Containing Packaged Subsistence Items: Project MQ 1-25:1. (Market Quality Research Div., Stored-Product Insects Branch, unpublished study; CDL:221615-Z.)

[The rate of application of DDVP was not reported in the following references.]

Shell Chemical Co. 1967. Determination of Vapona Residues in Stored Food Commodities Following Application of this toxicant. (Unpublished study submitted Apr. 6, 1967 under PP#7H2166 (221633). No MRID assigned.)

Shell Chemcial Co. 1967. Residues of DDVP in Packaged, Ready-to-Eat Food Commodities. (Unpublished study submitted Apr. 6, 1967 under PP#7H2166 (221633). No MRID assigned.)

Discussion of the data:

The U.S. Agricultural Marketing Service, Stored-Product Insects Branch (MRID 00056596) submitted data concerning residues in or on several packaged

commodities (flour in multiwall paper packages or in cotton sheeting; beans and rice in burlap bags; sugar in multiwall paper bags; raisins in corrugated boxes; dry milk in multiwall paper bags with polyethylene liners). Samples were placed in two 1,500 cu. ft. chambers and exposed to DDVP vapors from impregnated granules at 1.6-2.2 ug ai/l (2265-3115 g ai/50,000 cu. ft., or 22.7x to 31.1x the maximum registered rate).

The chambers were purged of DDVP after the 12 hour exposure period, and samples were taken for residue analysis at 0, 24, and 48 hours after exposure. Residues were highest in flour packaged in cotton sheeting (0.60 ppm at 0 hrs., 0.34 ppm at 24 hours, 0.27 ppm at 48 hrs.) and flour in paper (0.15 ppm at 0, hrs., 0.13 ppm at 24 hrs., 0.09 ppm at 48 hrs.). In all other commodities, residues were 0.01-0.08 ppm at 0 hrs., <0.01-0.18 ppm at 24 hrs., and <0.01-0.06 ppm at 48 hrs. Residues in beans, rice, sugar, and milk increased from the initial 0 hr. sample to the 24 hr. sample, probably as a result of DDVP leaching from the packaging material into the commodity. Residues in the burlap sacking containing rice (1,080 ug/sq. ft. at 0 hr.) were the highest of all packaging materials, and rice showed the greatest increase in residues from 0 hrs. (0.08 ppm) to 24 hrs. (0.18 ppm) of all commodities. Flour stored in cotton sheeting or multiwall paper containers showed a decline in residues between the 0 and 24 hr. sampling; residues in cotton sheeting (140 ppm at 0 hrs.) and multiwall paper (450 ppm at 0 hrs.) were the lowest of any commodity packaging materials. An adequate enzyme inhibition analytical method (Pr 5e-62) was used.

The U.S. Agricultural Marketing Service (MRID 00056595) submitted data concerning residues of DDVP in or on several packaged commodities (beans, flour [50 lb and 100 lb units], noodles, peanuts, raisins, rice, and sugar) stored in a 126,000 cu. ft. warehouse and subjected to 21 weekly applications of DDVP vapor at varying rates ranging from 7.1×10^{-4} to 16.7×10^{-4} g ai/ cu.ft.(0.4x to 0.8x the maximum registered rate). Samples for residue analysis were taken within 24 hrs. after the six-hour application period on weeks 1, 3, 5, 9, 13, 17, and 21; in addition, samples were taken immediately before application (i.e., 6 days after the previous weeks' treatment) on weeks 3, 9, and 17. For each commodity, samples were taken from the top

and bottom units of two-unit stacks, and consisted of the external and internal packaging surfaces, the surface layer of the commodity, and a blend of the commodity from the package. Residues were analyzed by an adequate enzyme inhibition method (Pr 5e-62).

Corrected residues were nondetectable (<0.1 ppm) in or on 199 samples of noodles, 199 samples of rice and 200 samples of raisins. Residues were nondetectable (<0.1 ppm) in 193 samples of beans; detectable residues of 0.10-0.15 ppm were found in six surface samples taken on weeks 17 and 21. Residues were nondetectable (<0.1 ppm) in 196 of 199 samples of sugar; detectable residues of 0.10-0.12 ppm were found in three surface samples taken on weeks 13 and 21. Residues in or on flour (50 and 100 lb lots) and peanuts are summarized in Table 7. Tolerance-exceeding residues occurred in several surface samples of flour and peanuts, but only in one blended sample of either commodity (peanuts, 24 hours after the 9-week application). Residues of DDVP were higher in the top units than in the bottom units. Residues were higher in samples of the commodity surface than in blended samples of the entire commodity. Residues were higher immediately following application compared with samples taken six days later. Residues tended to increase during the 21 weeks of the study. These results were reflected, as well, in the samples of the packaging material. In addition, residues were often many times higher on the outer surfaces of the top units compared with the bottom units. While residues on the outer surface of packaging materials often measured several hundred to several thousand ug/sq.ft., residues on the inner packaging materials measured no more than 72 ug/sq.ft. in any sample. With the exception of peanuts, which contain 36-48% fat (PAM, Vol. 1, Section 202.25), all of the commodities included in this study have low (<6%) fat content.

Shell Chemical Co. (MRID 00056593) submitted a study concerning residues of DDVP in or on packaged and unpackaged 1 lb. samples of salt pork, dry whole milk, powered cocoa, flour, and a single packaged 5 lb. sample of flour. The pork was wrapped in household plastic wrap, the milk was stored in a multiwall paper bag with a polyethylene liner, the flour was stored in a multiwall paper bag, and the cocoa was stored in a heavy paper can with

Table 7. DDVP residues (ppm) in or on packaged commodities after 1-21 weekly applications of DDVP in a 126,000 cu. ft. warehouse (MRID 00056595).

		Residues of DDVP (ppm)[c]					
		Flour, 50 lb		Flour, 100 lb		Peanuts	
Week	Unit	Commodity blend	Commodity surface	Commodity blend	Commodity surface	Commodity blend	Commodity surface
1[a]	Top	<0.10	<0.10-0.16	<0.10	<0.10-0.16*	<0.10-0.15*	0.32-0.89*
	Bottom	<0.10	<0.10	<0.10	<0.10*	0.10-0.30	<0.10-0.10*
3[b]	Top	<0.10	<0.10	<0.10	<0.10	<0.10-0.15	0.21-0.50
	Bottom	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
3[a]	Top	<0.10	<0.10-0.28	<0.10*	0.14-0.35*	0.13-0.31	1.00-1.63
	Bottom	<0.10	<0.10	<0.10	<0.10	<0.10-0.22	<0.10-0.31
5[a]	Top	<0.10	<0.10-0.23	<0.10	0.10-0.32	0.19-0.39	1.20-3.00
	Bottom	<0.10	<0.10	<0.10	<0.10	0.15-0.22	<0.10-0.30
9[b]	Top	<0.10	<0.10	<0.10	0.10-0.17	0.11-0.34	0.67-1.50
	Bottom	<0.10	<0.10	<0.10	<0.10	<0.10-0.23	<0.10-0.20*
9[a]	Top	<0.10	0.13-0.37	<0.10	0.22-0.35	0.17-3.90	1.00-2.50
	Bottom	<0.10	<0.10	<0.10	<0.10	0.16-0.36	0.27-1.80
13[a]	Top	<0.10	0.20-0.70	<0.10	0.56-0.76	0.50-1.00	2.00-5.40
	Bottom	<0.10	<0.10	<0.10	<0.10-0.12	0.24-0.87	0.87-1.80
17[b]	Top	<0.10	0.34-0.98	0.10	0.60-0.86	0.48-1.90	0.78-4.70
	Bottom	<0.10	<0.10	<0.10-0.10	<0.10-0.13	0.26-0.60	0.38-1.40
17[a]	Top	<0.10-0.10	0.28-0.75	<0.10-0.10	0.69-0.97	0.48-1.70	3.40-5.20
	Bottom	<0.10	<0.10	<0.10	<0.10-0.41	0.43-0.83	0.16-2.20
21[a]	Top	<0.10-0.13	0.68-1.50	<0.10-0.16	1.10-1.90	0.78-2.00	2.80-8.00
	Bottom	<0.10	<0.10-0.26	<0.10-0.11	<0.10-0.23	0.53-1.00	<0.10-5.00

[a]Sample taken within 24 hrs. after treatment.

[b]Sample taken just before treatment.

[c]Five samples analyzed in each test, except four samples in tests marked with *.

metal top and bottom. Unpackaged samples were placed in shallow paper trays. Duplicate samples of each packaged and unpackaged commodity were placed on the floor of two 1,500 cu. ft. chambers. Each chamber was treated with two applications of dichlorvos from aerosol cans: the first application consisted of 0.3 g dichlorvos; the second application, 2 hrs. later, consisted of 0.15 g dichlorvos (equivalent to 15 g ai/50,000 cu. ft. total application, 0.3 x the maximum registered rate). Samples of each commodity from each chamber were taken at 12 and 60 hrs. after the first treatment. Air was sampled and analyzed prior to the first treatment, and 0.5, 1, 2, 3, 4, 12, 12.5, 20, and 60 hrs. after the first treatment.

Residues of DDVP were higher in the unwrapped commodities than in the wrapped commodities, and were higher at 12 hrs. posttreatment than at 60 hrs. At 12 hr. posttreatment, residues in or on salt pork were 3.1-4.0 ppm vs 5.6-7.5 ppm in wrapped vs. unwrapped samples, respectively; in dry whole milk residues were 2.5-3.9 ppm vs. 5.0-7.1 ppm; in powdered cocoa residues were 1.2-2.6 ppm vs. 10 ppm (only one sample); in flour residues were 1.4-3.0 ppm vs. 3.0-5.0 ppm. At 60 hrs. posttreatment, residues in or on salt pork were 0.7-1.6 ppm vs. 2.5-4.0 ppm in wrapped vs. unwrapped samples, respectively; in dry whole milk residues were 0.9-1.1 ppm vs. 2.2-2.5 ppm; in powdered cocoa residues were 0.5-0.6 ppm; vs. 6 ppm (only one sample); and in flour residues were 0.6-0.8 ppm vs. 1.7-2.3 ppm. A sample of the 5 lb. packaged flour taken at 12 hrs. bore residues of 1.2 ppm, and a sample taken at 60 hrs. bore residues of 0.6 ppm. The paper packaging material itself was analyzed for DDVP residues and found to contain 385-400 ppm residues at 12 hrs., and 135-150 ppm at 60 hrs. Residues of dichlorvos in the air ranged from 2.5-5 ug/liter during the first 12 hrs. of the test. An adequate enzyme inhibition analytical method (Pr 5e-62) was used.

This study demonstrates that aerosol applications of DDVP, at approximately 0.3x the maximum rate, to nonperishable raw agricultural commodities having $\leq$6% and >6% fat content will result in tolerance-exceeding residues. No data reflecting residues at maximum permissible aerosol use rates are available. Therefore, these data are needed to determine maximum residues resulting from maximum use rates.

Tobacco

Use directions and limitations:

The following formulations may be used by professional pest control operators at the rates and under the conditions specified on stored, unfinished tobacco:

Formulation[a]	Rate[b]	Use and limitations
SAI		
1.16 lb/gal EC	50-100 g	Space treatment in closed warehouses storing unfinished tobacco only. Dilute in odorless oil or other non-flammable oil solutions known to be safe for use in tobacco warehouses. Repeat as needed.
1.59 lb/gal EC	50-100 g	
2 lb/gal EC	50-100 g	
4 lb/gal EC	50-100 g	
4.48 lb/gal EC	50-100 g	
1.15 lb/gal SC/L	50-100 g	
8.39 lb/gal SC/L	50-100 g	
10 lb/gal SC/L	50-100 g	
50% SCL	50-100 g	
100% SCL	50-100 g	
MAI		
5.38 lb.gal EC	50-100 g	
5% RTU	40-80 fl.oz	
SAI		
1.9 lb/gal RTU	25-50 g	Space treatment. For use only with timed and metered aerosol equipment. Treated area must be posted at entrances for a period of 8 hours. Air-conditioned or forced-air ventilated areas must be posted for 2 hours, or until concentration of DDVP in air is <1 mg/m^3. Dispense only between 5 pm and midnight, or between 5 pm and 4 am for air-conditioned areas.
5.8 lb/gal RTU	25-50 g	
SAI		
10% RTU	8 fl.oz/ 24,000 cu.ft.	Space treatment by thermal fog generator only. Length of exposure is 2 hours with windows and doors closed, then ventilate at least 1 hour before reentering.
15% RTU	8 fl.oz/ 24,000 cu.ft.	

Continued.

Formulation[a]	Rate[b]	Use and limitations
MAI		
50% SC/L	50-100 g	
0.74 lb/gal RTU	25-50 g	
2.2 lb/gal RTU	25-50 g	
5% RTU	25-50 g	
10% RTU	25-50 g	
10% RTU	33.3-66.6 fl.oz.	
SAI		Space treatment to be applied between 5 pm and midnight. Treated area should not be reentered for 4 hours or until particles have settled.
20% PrL	25-50 g	
40% PrL	25-50 g	
SAI		For use against the cigarette beetle by aerosol or thermal fogging applicator. For use only on stored tobacco hogsheads or cases in warehouses.
0.8 lb/gal EC	49.2-49.3 g	
1.13 lb/gal EC	49.2-49.3 g	
2.2 lg/gal EC	49.2-49.3 g	
SAI		For use against the tobacco moth by aerosol or thermal fogging applicator. For use only in stored tobacco in hogsheads or cases in warehouses.
0.9 lb/gal EC	98.5-98.6 g	
1.13 lb/gal EC	98.5-98.6 g	
2.2 lb/gal EC	98.5-98.6 g	

[a] SAI - single active ingredient. MAI - multiple active ingredient, with 1,1,1-trichloroethane.
[b] Weight active or volume of formulation per 50,000 cu.ft., except where noted.

Conclusions:

No data were submitted concerning residues of DDVP in or on tobacco. Use of a pesticide on tobacco does not require a tolerance or an exemption from the requirement to obtain a tolerance. Nonetheless, data are needed to assess the use of the tobacco. The data required include a residue profile for the tobacco and its smoke. Data from the following studies must be submitted to show conclusively the level of residue likely to result from the use of the pesticide:

- o Total residues on stored tobacco following multiple space treatments with an EC or SC/L formulation of DDVP at 100 g ai/50,000 cu. ft. If residues levels of 0.1 ppm or greater are detected, pyrolysis products derived from the active ingredient must be characterized and the level of residue in smoke must be quantified.

References:

N/A.

Discussion of the data:

N/A.

MAGNITUDE OF THE RESIDUE IN COOKED FOOD

The data in this section are presented for informational purposes only.

References (used):

00042707. Shell Chemical Co. 1962. Determination of Vapona Insecticide Residues in Rice, Flour, Gravy, and Biscuits following Application of this toxicant: RES-62-10. (Unpublished study received Apr. 18, 1962 under unknown admin. no.; CDL:108822-A.)

Discussion of the data:

Shell Chemical Co. submitted data from a single study (MRID 0042707) concerning residues of DDVP in rice, flour, gravy, and biscuits following cooking. Two lots of rice were spiked with 4.5 and 19 ppm DDVP, respectively, and two lots of flour were spiked with 4.5 and 14 ppm DDVP, respectively. The rice was cooked by boiling 20-30 min in water. Treated flour was used to make biscuits and gravy; biscuits were cooked 10-12 min at 450 F, and gravy was boiled 2 min. After cooking and cooling to room temperature, rice, biscuits, and gravy were analyzed for residues by an adequate enzyme inhibition analytical method (MMS-30/60). Residues in cooked rice were 0.1 and 0.4 ppm (at 4.5 ppm and 19 ppm fortification levels, respectively), residues in biscuits were 0.4 and 1.8 ppm (at 4.5 and 14 ppm fortification levels, respectively), and residues in gravy were 0.1 and 0.4 ppm (at 4.5 and 14 ppm fortification levels, respectively). Residues in untreated, uncooked, and cooked samples of rice, flour, biscuits, and gravy were nondetectable (<0.1 ppm). Recovery from fortified samples of rice, flour, biscuits, and gravy was 80-100%.

MAGNITUDE OF THE RESIDUE IN MEAT, MILK, POULTRY, AND EGGS

Tolerances:

Separate tolerances of 0.02 ppm (negligible residues) have been established for residues of DDVP in the fat, meat, and meat by-products of cattle, goats, horses, and sheep, and in milk [40 CFR 180.235(a)]. Separate tolerances of 0.05 ppm (negligible residues) have been established for residues of DDVP in the fat, meat, meat by-products, and eggs of poultry [40 CFR 180.235(a)]. A tolerance of 0.1 ppm (negligible residues) has been established for residues of DDVP in the edible tissues of swine [21 CFR 561.180; 40 CFR 180.235(b)], covering both its use as an anthelmintic in swine feed and as an insecticide applied directly to swine.

NOTE TO P.M. - At the present time, tolerances for residues of naled and DDVP, expressed as naled, in milk and the fat, meat, and meat byproducts of cattle, goats, hogs, horses and sheep are established at 0.05 ppm (40 CFR 180.215). Thus, the following should be added to 40 CFR 180.3, pending receipt of the requested livestock feeding studies for both naled and DDVP:

> Where tolerances are established for residues of both DDVP (40 CFR 180.235), and naled and DDVP (40 CFR 180.215), in or on the same raw agricultural commodity, the total amount of such pesticides shall not yield more residue than that permitted by the higher of the two tolerances.

Use directions and limitations:

Cattle, Goats, Horses, Sheep, and Swine: Many formulations are registered for use in a wide variety of applications. Refer to the EPA Index to Pesticide Chemicals for 2,2-dichlorovinyl dimethyl phosphate for details. Direct insecticidal applications are permitted on cattle, goats, horses, sheep, and swine, at a maximum rate of 2 fl. oz. of a 1% finished spray (equivalent to 591 mg ai) per animal per day on beef and dairy cattle and horses, and 1 fl. oz. of a 1% finished spray (equivalent to 296 mg ai) per animal per day on goats, sheep, and swine. Do not treat Brahman cattle or Brahman crosses. Do not apply regularly to calves under six months of age. A 20 min premilking interval is in effect. There is no preslaughter interval.

DDVP also is registered for use as an anthelminitic in swine feed at the following rates [21 CFR 520.600(e)(2)]:

Weight of animal in pounds	Milligrams ai/animal	Milligrams ai/pound body weight
20-30	189	6.3-9.4
31-40	252	6.3-8.1
41-60	378	6.3-8.1
61-80	454	5.7-7.4
81-100	567	5.7-7.0
Adult gilts, sows, and boars	567	<5.7

Do not use simultaneously or within a few days before or after treatment with or exposure to cholinesterase inhibiting drugs, insecticides, or chemicals. Swine may be retreated in 4 to 5 weeks.

Poultry: DDVP may be used for direct animal treatment, premise treatment when animals are present, premise treatment after animals have been removed, and manure treatment. There is no preslaughter interval.

The 5.7% EC is registered for direct animal use on caged laying chickens. Mix 1 pt formulation in 6 gal water and apply at 1-1.28 fl. oz./bird (equivalent to 35 to 45 mg ai/bird) to the vent and fluff areas from below. Repeat as necessary every 14 days.

The 20% impregnated material is registered for continuous use at the rate of 1 strip/row of cages of laying chickens.

The 25% RTU is registered for premise treatment to empty cages. Spray entire interior of coop and house thoroughly. Repeat at 14 day intervals.

The 1% PrL is registered for premise treatment with birds present in caged

laying houses equipped with aerosol piping system and automatic timer. Apply at the rate of 0.59 sec/1,000 cu. ft. with curtains closed, or at the rate of 10 sec/1,000 cu. ft. with curtains open. Make only one application daily. Do not place nozzle within 5 feet of food, water, or fowl.

The 5.7% EC is registered for premise treatment with broiler chickens present. Mix 1 pt. formulation/6 gal water and apply 1-2 gal/1,000 sq. ft., thoroughly spraying walls, roosts, cracks, and crevices. Spray birds lightly.

The 15% impregnated material is registered for use as a leg band on turkeys at the rate of 1 band/bird. Apply 5-6 weeks before intended marketing. Remove band before processing.

The following formulations are registered for use as manure treatments beneath cages or perches as a coarse wet spray that may be repeated every 7 days: i) 0.5 and 1% G, 0.02 oz./100 sq. ft., or 0.13 oz./gal at 0.5 gal/100 sq. ft.; ii) the 2 lb/gal EC as a 0.5% finished spray at 0.5 gal/100 sq. ft.; iii) 1 gal of the 1.5% EC/20 gal water; iv) 0.01 oz. of the 0.25% G/100 sq. ft.; v) 1 bait cake of the 0.25% P/T per dropping pit; vi) the 0.5 lb/gal EC as a 0.25% finished spray; and, vii) 1 gal of the 3.2% EC/48 gal water.

The maximum expected dietary intake of DDVP by dairy and beef cattle is 1.25 ppm, based on a diet of 10% soybeans, 20% oats, 20% cottonseed, and 50% alfalfa hay. [The tolerance for residues in or on packaged or bagged nonperishable raw agricultural commodities (RACs) containing >6% fat (which includes soybeans, oats, and cottonseed) is 2 ppm. The tolerance for residues in or on bulk, stored nonperishable RACs, regardless of fat content (which includes alfalfa hay) is 0.5 ppm.] The maximum expected intake by swine is 2 ppm based on a diet consisting of 50% oats, 20% soybeans, 10% cottonseed, and 20% flax seed (>6% fat).[Also, DDVP is ingested by swine via its use as an anthelmintic - see above.] For poultry, the maximum expected intake of DDVP residues is 1.55 ppm based on a diet composed of 50% soybeans, 20% oats, and 30% corn (<6% fat).

Conclusions:

The available data indicate that no measurable residues of DDVP will occur in the meat, fat, and meat by-products of cattle, goats, horses, sheep, and poultry and in milk and eggs following direct application to animals. However, no data regarding direct spray applications to swine are available. Also, no feeding studies are available regarding the potential for residues occurring in the tissues, and milk and eggs of livestock as a result of ingestion of feed bearing residues of DDVP. Thus, the following additional data are required:

- Residues of DDVP must be determined in the fat, muscle, liver, and kidney of ruminants sacrificed 24 hours after 28 consecutive days of ingestion of 1.3 ppm (1x), 3.8 ppm (3x), and 13.0 ppm (10x) of DDVP in the total diet (dry weight basis). Residues must also be determined in milk collected twice daily throughout the feeding period.

- Residues of DDVP must be determined in fat, muscle, liver and kidney of poultry sacrificed 24 hours after 28 consecutive days of ingestion of 1.6 ppm (1x), 4.8 ppm (3x), and 16.0 ppm (10x) of DDVP in the total diet (dry weight basis). Residues must also be determined in eggs collected twice daily throughout the feeding period.

- Residues of DDVP must be determined in the muscle, liver, kidney, and fat of swine fed 28 consecutive days with 2 ppm (1x), 6 ppm (3x), and 20 ppm (10x) of DDVP in the total diet (dry weight basis) and orally dosed once at 6 mg/lb body weight (anthelmintic use) on the 28th day. Animals must be sacrificed 24 hours after the final dose.

- Swine must be mist sprayed with 1 fluid ounce of a 1% ai finished spray per day for 28 consecutive days and sacrificed within 24 hours after the final treatment. Residues must be determined in muscle, liver, kidney, fat and skin.

References (used):

00115945. Shell Chemical Co. 1973. Residue Information in Support of Vapona Insecticide Residual Wall Spray Emulsifiable. (Compilation; unpublished study received on unknown date under 201-EX-45; CDL:238131-A.)

00116436. Shell Oil Co. 1962. Letter sent to L. Lykken dated Feb. 9, 1962 Results of tests: Valpona residue in cows milk. (Unpublished study received Feb. 23, 1963 under 201-164; CDL:120488-A.)

00118639. Koos, B. 1973. Determination of DDVP Residues in Chicken Eggs and Tissues following Direct Application with Vapona Insecticide Residual Wall Spray Emulsifiable: TIR-26-009-73. (Unpublished study received on unknown date under 201-EX-45; submitted by Shell Chemical Co.; CDL:238130-A.)

00119537. Shell Chemical Co. 1970. The Results of Tests on the Amount of Residues Remaining, Including a Description of the Analytical Methods Used: Vapona. (Compilation; unpublished study received Nov. 5, 1970 under 1F1059; CDL:093372-C.)

00139843. Marymiller, R.L.; Bramhall, E.L. 1971. Determination of Vapona (R) Insecticide Residues in Turkeys following Exposure to This Insecticide via Resin Strip Leg Bands: TIR-24-500-71. (Unpublished study received Jan. 30, 1973 under 201-341; submitted by Shell Chemical Co.; CDL:001044-G.)

00139844. Brady, U.E. 1972. Determination of Vapona Insecticide Residues in Turkeys after Exposure to Vapona Leg Bands. (Unpublished study received Jan. 30, 1973 under 201-341; prepared by Univ. of Georgia, Dept. of Entomology, submitted by Shell Chemical Co.; CDL:001044-I.)

References (not used):

[The following references are redundant with previously cited MRIDs.]

00049085. Shell Chemical Company. 1971. Residue Study: Vapona on Poultry.

(Compilation; unpublished study received unknown date under 201-341; CDL:222759-A.)

00116502. Shell Chemical Co. 1965. Study: Vapona Residues in Chicken Eggs & Poultry Tissue. (Compilation; unpublished study received May 17, 1966 under 201-302; CDL:028514-B.)

00116870. Shell Chemical Co. 1970. The Results of Tests on the Amount of Residues Remaining, Including a Description of the Analytical Methods Used: Vapona. (Compilation; unpublished study received Aug. 12, 1971 under 1F1059; CDL:091894-I.)

00117260. Shell Chemical Co. 1965. Vapona: Residues in Poultry--Tissue and Eggs. (Compilation; unpublished study received May 17, 1966 under 201-158; CDL:120049-A.)

00117684. Page, C.; Hobbs, J. 1962. Results of Analysis on Eggs and Chicken for DDVP ... Residue. (Unpublished study received Mar. 9, 1962 under unknown admin. no.; prepared by Science Assoc., Inc., submitted by Chemspex, Inc.; CDL:120062-A.)

00117688. Shell Chemical Co. 1962. Determination of Vapona Insecticide Residues in Chickens & Eggs following Applicatiion of This Toxicant: RES-61-41. (Unpublished study received Aug. 8, 1962 under unknown admin. no.; CDL:120069-A.)

00117689. Chemspex, Inc. 1962. Report of Analyses on DDVP and Vapona Insecticide Residues in Various Products. (Compilation; unpublished study received Oct. 8, 1962 under 7221-1; CDL:120070-A.)

00118168. Shell Chemical Co. 1971. Vapona: Residues in Poultry. (Compilation; unpublished study received Apr. 1, 1972 under 201-EX-43; CDL:122725-C.)

00139841. Shell Chemical Company. 19?? Residue Data from Dr. C.B. Nelson, Fillmore, California. (Unpublished study received Jan. 30, 1973 under 201-341; CDL:001044-E.)

Discussion of the data:

Cows: Shell Chemical Co. (MRID 00115945) submitted a study concerning residues of DDVP in tissues and milk from three lactating Holstein dairy cows resulting from daily mist spray application of 2 oz. per animal of a 1% DDVP spray solution (1x the maximum registered rate) for 31 consecutive days. Applications were made each morning after milking. Milk was sampled for residue analysis from three experimental cows and one control cow prior to the start of the experiment, 2 hrs after the first spray application and on days 1, 2, 4, 8, 16, 24, and 31 of the test. Morning and evening milk were combined and subsampled for analysis. All cows were slaughtered one day after the final application and samples of fat (renal, omental, and subcutaneous), liver, muscle, kidney, and blood were placed in polyethylene bags and frozen prior to residue analysis. Residues were nondetectable (<0.01 ppm) in all 64 milk and tissue samples. Residues were analyzed by an adequate gas chromatographic analytical method. Recoveries from an unspecified number of samples fortified at 0.01 ppm (the presumed limit of detection) were 93-97% from milk, 80-85% from muscle and fat, and 86% from blood.

Included in the same MRID is a study concerning residues of DDVP in the milk of Guernsey and Holstein cows each treated twice daily with 2 fl. oz. of 1% or 0.5%, respectively, of DDVP spray solution (mixed with 1% Ciodrin). Milk for residue analysis was sampled daily for 28 days and frozen prior to analysis. Results were presented for the final samples taken on day 28 after 55 spray treatments. Residues were "nil" (<0.1 ppm) in seven samples. Residues were analyzed by an adequate enzyme inhibition method (MMS-30/63), however, the reported limit of detection (0.1 ppm) is above the established tolerance (0.02 ppm). Therefore, these data cannot be used to assess the tolerance.

Shell Oil Co. (MRID 00116436) submitted a study concerning residues of dichloroethanol (DCE), a presumed metabolite of DDVP, in milk from dairy cows sprayed 0, 10, 17, and 37 times with DDVP at 2 oz. of 1% spray per cow. Residues of DCE in four samples from treated cows and one from an untreated cow were nondetectable (<0.05 ppm). Residues were analyzed by a poorly described GLC method. Recovery from fortified milk was claimed to be 90%. DCE is not

presently considered a residue of concern resulting from application of DDVP. This study is presented for informational purposes only.

Poultry: Shell Chemical Co. (MRID 00119537) submitted the results of eight studies concerning residues of DDVP in the eggs and tissues of laying chickens following direct application of DDVP. In four tests, single spray applications were made at rates of 4-295 mg/bird (0.1x to 6.6x the maximum registered rate). Eggs were sampled at intervals, and chickens were killed for tissue analysis at intervals following application through 28 days posttreatment.

Eggs were frozen prior to analysis. Chickens were processed by commercial methods and fat, skin, liver, gizzard, and meat were frozen prior to analysis. Residues in 39 eggs ranged from nondetectable (<0.02 or <0.03 ppm) to 0.03 ppm. Residues in 76 tissue samples ranged from nondetectable (<0.02 to <0.05 ppm) to 0.08 ppm; eight samples from chickens treated at the highest rate (6.6x) bore detectable residues, and only one (skin, six hrs post treatment, 0.08 ppm) exceeded the tolerance. Residues in 28 untreated control samples were non-detectable (<0.02 to <0.05 ppm). Residues were analyzed by an adequate GC analytical method (PMS-G-913/69). Recovery from 37 samples of eggs and tissues fortified at 0.05-0.2 ppm was 50-120%; however, recovery from 11 samples of liver fortified at 0.1-0.2 ppm was only 40-90% (62% average).

In another test, two groups of chickens were treated daily for 30 consecutive days with an aqueous spray of DDVP at 2.8 or 11.3 mg ai/bird. Eggs were collected and composited into three weekly samples for analysis. Residues in six composited egg samples were nondetectable (<0.02 ppm). A single untreated control sample bore 0.02 ppm residues. An adequate enzyme inhibition analytical method (MMS-30/64) was used. Recovery from three samples fortified at 0.1 ppm was 90-100%. This test does not conform to registered uses, which do not permit daily direct animal treatments.

Results also were presented from two studies in which 20% impregnated cords or strands were suspended beneath cages at the rate of 1 foot of cord per 1 foot of cage. It was not stated in the test how many chickens were held in each

cage. Eggs and chickens were removed for residue analysis at 1 to 5 days. Eggs and tissues were frozen prior to analysis. Residues in eight samples of eggs (including 2 composite samples of 12 eggs each) were nondetectable (<0.02 to <0.05 ppm). Residues in six samples of meat, fat, and liver also were nondetectable (<0.03 ppm or <0.05 ppm). Residues in 16 untreated control samples were non-detectable (<0.02 to <0.05 ppm). These studies are inadequate to assess the established tolerances because they were not continued for a sufficient length of time.

Submitted in this same MRID (00119537) were the results of five tests in which 2% G or a 1% finished spray (made from an unspecified formulation) were applied to the floor beneath cages at rates of 3.6-36.1 g ai/100 sq. ft. (approximately 3.3x to 32.8x the maximum registered rate). Eggs were removed for residue analysis and chickens were slaughtered for tissue residue analysis at intervals from 1 to 29 days posttreatment. Residues in 74 samples and 20 untreated samples of eggs, meat, liver, and fat, were nondetectable (<0.02 to <0.05 ppm). Adequate enzyme inhibition analytical methods (MMS-30/60 and MMS-30/64) were used.

Shell Chemical Co. (MRID 00118639) submitted the results of a study in which caged chickens were sprayed with a 2% solution of DDVP at a rate of approximately 50 mg/bird (1.1x the maximum registered rate). Beginning two days posttreatment and continuing until 10 days posttreatment, eggs were collected and composted for residue analysis. At two days posttreatment, nine chickens were slaughtered for tissue residue analysis. Residues in 66 eggs from hens following treatment and on an uncertain number of eggs from untreated hens ranged from nondetectable (<0.01 ppm) to 0.02 ppm. Residues in 46 samples of muscle, heart, liver, fat, and ova (eggs inside the bodies of slaughtered hens) from treated and untreated hens ranged from nondetectable (<0.01 ppm) to 0.02 ppm. An adequated GLC analytical method (a modification of MMS-R-222-1) was used for residue analysis. Recovery from 19 samples fortified at 0.025, 0.05, or 0.1 ppm was 70-130%.

Shell Chemical Co. (MRIDs 00119537, 00139843, and 00139844) submitted the results of five tests concerning residues in tissues of turkeys and the tissues and eggs of chickens resulting from continuous exposure to DDVP from impregnated

resin leg bands. Bands of 15% impregnated material were applied at the rate of one to four per turkey (MRIDs 00139843 and 00139844) and left in place 14-50 days prior to slaughter. We believe this adequately represents the maximum registered rate, which is 1 band of the 20% impregnated material per turkey 5-6 weeks prior to slaughter. Residues were analyzed using adequate GLC analytical methods (MMS-R-201-1 and MMS-R222-1). Recovery data were not provided, however.

In the test on chickens (MRID 00119537), one leg band of the 20% impregnated material was attached to each bird. Eggs were collected for residue analysis at 1, 3, 5, 7, 14, 21 and 28 days after attachment. Chickens were killed for tissue residue analysis 1, 3, 28 days after the leg bands were attached. Residues in 13 egg, six meat, fat, and liver, and 14 untreated control samples, were nondetectable (<0.03 or <0.05 ppm). Residues were analyzed using an adequate GLC method (PMS-G-913/69). Leg bands are not registered for use on chickens.

Shell Chemical Co. (MRID 00119537) also submitted the results of tests reflecting direct bird treatments with a 1% and a 5% D formulation and a test in which a 5% D was added to the diet of chickens. These formulations are not registered for use on poultry. In addition, the sensitivity of the analytical method used in these tests (MMS-30/60) was reported to be 0.1 ppm, which is greater than the established tolerances.

FOOD HANDLING ESTABLISHMENTS

Tolerance:

No tolerances or regulations have been established covering residues of DDVP in or on foods resulting from treatment of food handling establishments.

Use directions and limitations:

DDVP formulations are registered for use in indoor commercial, institutional, and industrial areas (directed spray, space treatment, bait - liquid and dry, and insecticidal strip), indoor eating establishments (crack and crevice, spot treatments and baits), food markets (directed spray, space treatments, and

baits), and food processing, handling and storage plants/areas (crack and crevice, space treatment, and baits). Rates of use for EC, SC/L, PrL, RTU, Impr, and G (bait) formulations are expressed in a variety of ways [% ai spray, oz/ft^3, g/ft^3, sec/ft^3 (PrL), oz/ft^2, $strips/ft^2$, etc.].

Conclusions:

No data are available concerning possible residues of DDVP in or on food items resulting from the registered food handling establishment uses. The following data are required:

- o (i) Food service establishments (restaurants and groceries) must be treated via space treatment (PrL); (ii) food manufacturing establishments (two types) must be treated via space treatment (SC/L), and resin strips; and (iii) food processing establishments (two types) must be treated via space treatment (PrL). Treatments must be made at maximum registered rates. The registrant must determine, from the above applications, residues of concern from direct deposition of spray droplets on food, volatilization and subsequent absorption by foods, and all other possible modes of exposure of foods and residue transfer routes as outlined in the EPA Pesticide Assessment Guidelines, Subdivision O, Residue Chemistry, 171-04(C)(5). Representative food samples must be examined for residues of concern from these possible modes of exposure; the food must include an oily food (eg., butter), baked cereal products, beverages, raw and processed meats, and fresh fruits and vegetables (lettuce). Tests must also include conditions of possible misuse of DDVP formulations, e.g., applying an exaggerated rate or direct application of DDVP to exposed foods (Refer to EPA Pesticide Guidelines for details of the type of data required). In addition, air samples must be analyzed in food manufacturing establishments in which Impr formulations (resin strips) are used to determine the concentration of DDVP residues in air.

On receipt of these data, appropriate tolerances and regulations must be established.

REGULATORY INCIDENTS

No information has been received to date from USDA pertaining to regulatory incidents involving DDVP although the Registration Division (OPP, EPA) has requested such information. The following information has been received pertaining to DDVP residues in or on food commodities tested through FDA's residue monitoring program [letter to G.T. LaRocca (EPA) from E.L. Gunderson (FDA), dated 1/22/86].

Between 1978 and 1985, FDA analyzed 40,000 samples of domestic and imported foods analyzed by methods capable of either fully or partially recovering DDVP from non-fatty foods. Results of surveillance monitoring revealed the presence of measurable residues in one sample of domestic chili pods (1979; 5.07 ppm), four samples of domestic rice products (1980; 0.07, 0.06, and 0.01 ppm and "trace"), one sample of domestic strawberries (1980; 0.1 ppm), two samples of domestic shelled peanuts (1982; 0.03 and 0.03 ppm) and one sample of imported tomatoes (1980; 0.03 ppm). [It should be noted that no tolerances or registered uses for DDVP on chili pods or strawberries exist.]

TOLERANCE REASSESSMENT SUMMARY

It should be noted that data daps exist for the storage stability of DDVP in plants and animals. Because the requested data and perhaps the continued adequacy of all established tolerances are dependent upon the results of the above studies, it is imperative that the storage stability data be collected prior to the requested residue data. Furthermore, the conclusions stated below are subject to change on receipt of the requested storage stability data.

Insufficient data are available to ascertain the adequacy of the established tolerances for residues of DDVP in or on radishes, lettuce, tomatoes,

cucumbers, milk, eggs, and the meat, fat, and meat by-products of cattle, goats, hogs, horses, sheep, and poultry. A processing study is required for tomatoes and data reflecting use of DDVP on stored, unfinished tobacco are also required. Tolerances for DDVP residues in or on figs, dried figs, and mushrooms are adequately supported.

The tolerances for residues of DDVP in or on mushrooms, tomatoes, cucumbers, and lettuce should be expressed as DDVP per se rather than naled, as currently stated in 40 CFR 180.235. DDVP labels permitting use on mushrooms, tomatoes, lettuce, and cucumbers must be amended to prohibit use of naled on crops treated with DDVP (refer to p. 39 for details). Also, 40 CFR 180.3 must be amended as described under Tolerances on p. 83.

Insufficient data are available to ascertain the adequacy of the established tolerances for residues of DDVP in or on bulk stored nonperishable raw agricultural commodities and packaged or bagged nonperishable raw agricultural commodities and processed food. Also, a food additive tolerance or regulation is needed regarding the use of DDVP in food handling establishments.

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